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A Study of Physical Properties

BONDiT™ B-45TH Epoxy

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1. Introduction

A study of the physical and mechanical properties of B-45TH and B-45 was carried out in order to better understand potential applications of the two-part epoxy system.

TMA, TGA and DSC were used to study the effect of curing method and mixing technique on several important physical property characteristics. Table 1 summarizes the experiments that were performed.

Analysis of the physical properties of B-45TH included for the resin mixed with a static mixer[1] and by hand mix[2]:

- Comparing the glass transition temperature of a hand mixed Vs a static mixed sample,
- The outgassing behavior of the epoxy using three curing methods and outgassing under elevated operational temperatures,
- The extent of curing reaction as a function of curing method,
- Environmental test of 85RH / 85°C with three curing methods,
- Moisture absorption, (hand mix)
- Oil absorption, (hand mix)
- Percent solid of hand mix epoxy compared to a static mix,
- Contact angle relative to static mix and hand mix,
- Joint strength relative to static mix and hand mix.

Other tests included:

- Tensile strength, (hand mix)
- Static fatigue strength, (hand mix)
- Tear strength, (hand mix)
- Velocity of sound, (hand mix)
- Thermal conductivity, (hand mix)
- Coefficient of thermal expansion, (hand mix)
- Lap shear tests on various substrates, (static mix)
- Peel tests on various substrates, (static mix)
- Bead length Vs bead width for B-45TH dispensed from a static mixer.

[1.] Static mix = parts A & B measured 2:1 by a double-barreled cartridge dispensed with a plunger in a hand actuated gun through a 6" helical 21 element static mixer tube having a 1/16" exit nozzle.

[2] Hand mix = parts A & B measured in 2:1 ratio by weight and mixed by hand.

Table 1. Summary of experiments performed on B-45TH epoxy to examine some key physical properties.

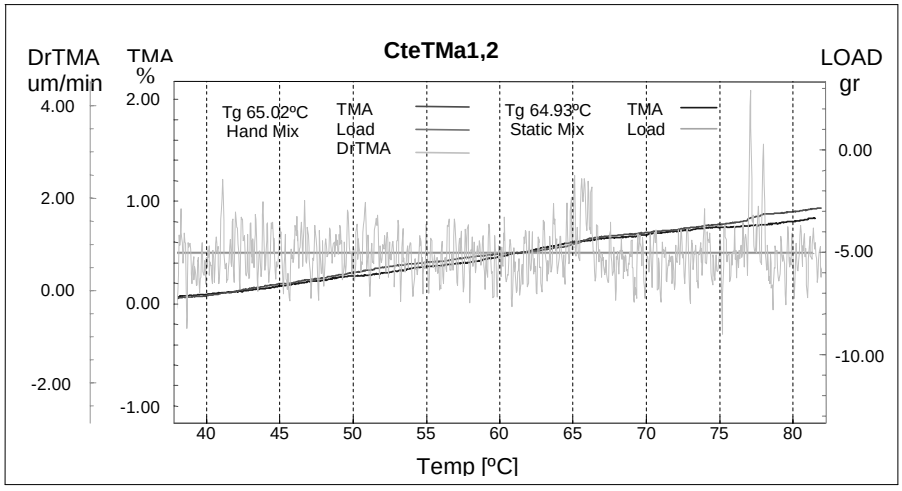
Test#	Curing method	Experiment	Thermal Analysis Cycle	Comments
Glass Transition Temperature		determine if there is a difference in Tg	All runs	
TgDSC1	cure for 68 hrs @ RT	by using different mixing methods	25-300°C, 10°C/min	mix with static mixer
TgDSC2	cure for 68 hrs @ RT			mix by hand
CteTMA1	cure for 72 hrs	second attempt to determine	All runs	mix with static mixer
CteTMA2	cure for 72 hrs	Tg	25-200°C, 2.°C/min	mix by hand
Outgassing		volatiles and water as a function of curing method	check for outgassing of All runs	
OgTGA1	cure for 24 hrs @ RT		25-300°C, 10°C/min	cure set 95%
OgTGA2	cure for 30 min @ RT then 4 hrs at 65C			cure set 98%
OgTGA3	cure for 30 min @ RT then 2 hrs at 93C			cure set 98%
Curing Tests		measure extent of completion of curing reaction for the three curing methods		all runs
CtDSC1	cure for 24 hrs @ RT		25-300°C, 10°C/min	cure set 95%
CtDSC2	cure for 30 min @ RT then 4 hrs at 65C			cure set 98%
CtDSC3	cure for 30 min @ RT then 2 hrs at 93C			cure set 98%
CtDSC4	freshly mixed, no cure			
Environmental Study (humidity)		determine the amount of water absorption of the epoxy cured using the three recommended methods		all runs
EsTGA4	cure for 24 hrs @ RT		25-300°C, 10°C/min	cure set 95%
EsTGA5	cure for 30 min @ RT then 4 hrs at 65C			cure set 98%
EsTGA6	cure for 30 min @ RT then 2 hrs at 93C			cure set 98%
Mixing Effects		check solids content of epoxy		all runs
MeTGA1	wet hardener	components and epoxy	550°C, 30 min hold, 10°C/min	ash hardener (wet)
MeTGA2	wet resin			ash resin (wet)
MeTGA3	freshly mixed, no cure			ash mixture (static mixer)
Me TGA4	freshly mixed, no cure			ash mixture (hand mix)
Contact Angle		determine the difference in surface	all runs: no thermal analysis was performedThe contact angle geoniometer was used for measuring the contact angle for the epoxy	
Series Cas1,2	cured for 96 hrs @ RT	energy of static and hand mixed epoxy was performed		mixed 20 samples
Series Cah1,2	cured for 96 hrs @ RT	also, difference between epoxy side exposed to air during curing vs unexposed side		hand mixed 20 samples
Joint Strength		determine if the joint strength from a static mixed vs a hand mixed sample population are comparable	all runs: no thermal analysis was performed A Chatillon joint strength machine was used	
Series Jss1,2	cured for72 hrs @ RT			static mixed 20 samples
Series Jsh1,2	cured for 72 hrs @ RT			hand mixed 20 samples

2. Glass Transition by TMA Method

The ***glass transition*** temperature was not apparent in either the epoxy that was hand mixed or sample that was mixed using a static mixer by analyzing with the DSC. The TMA was then used to determine the ***Tg*** for the two samples.

Theoretically the ***glass transition*** temperature should be approximately 60°C. Expanding the graphs from 40°C to 80°C did reveal that the ***Tg*** for the static mixed sample is 64.93°C and the ***Tg*** for the hand mixed sample is 65.02°C. These values are in agreement with the theoretical ***Tg***. The difference in ***Tg*** between the two samples is negligible.

Figure1. Glass transition profiles

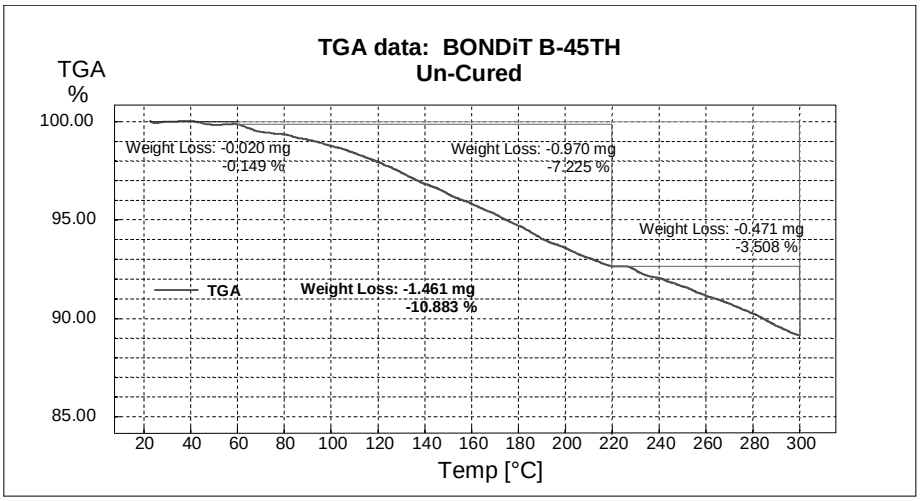


3. Outgassing

Thermogravimetric Analysis (TGA) was used for the outgassing study.

The initial outgassing study was performed on un-cured material from room temperature to 300°C at a rate of 10°C/min. This scan determines the amount of ***outgassing*** during cure as a function of weight loss. At 60°C, the weight loss due to ***outgassing*** is only 0.149%. Up to 220°C, were curing appears to end and before degradation, the total weight loss is 7.225%.

Figure 2. Uncured B-45TH Outgassing



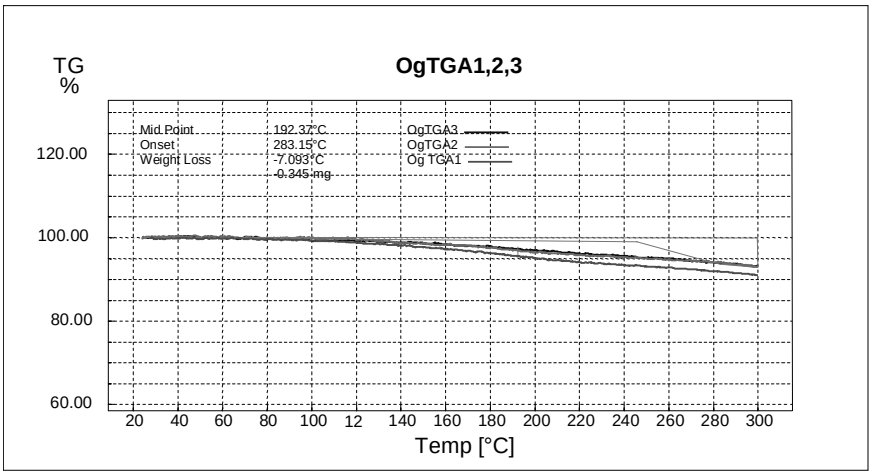
Further testing by TGA indicates a correlation between weight lost during the thermal cycle and curing technique. In Figure 3 and Table 2 the results are summarized.

Table 2. Percent weight loss as a function of curing method

Test #	Curing Method	Onset of Outgassing	% wt. loss
OgTGA1	RT= 23.5 hrs	80°C	9.047
OgTGA2	65°C for 4 hrs	140°C	7.093
OgTGA3	93°C for 2 hrs	120°C	6.93

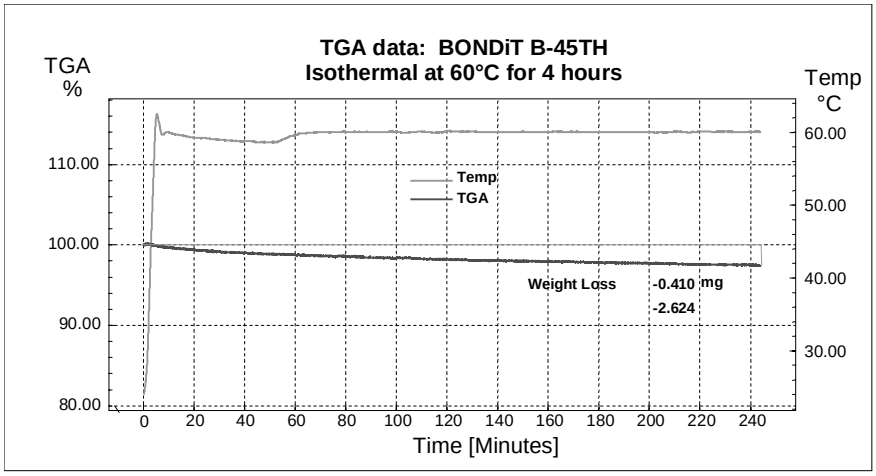
From the results, it is possible to conclude that when cured under lower temperatures there is more free water and volatile material in the epoxy. There also is a significant difference between the onset of outgassing temperature for the heat-cured methods and the sample cured at ambient temperature.

Figure 3. Graph of epoxy outgassing profiles



In order to know the amount of outgassing at regular operating temperatures, an isothermal scan at 60°C for four hours was performed and only 2.624% weight loss was measured. This relates to 0.66% ***outgas*** weight loss per hour.

Figure 4. Isothermal Cure Weight Loss

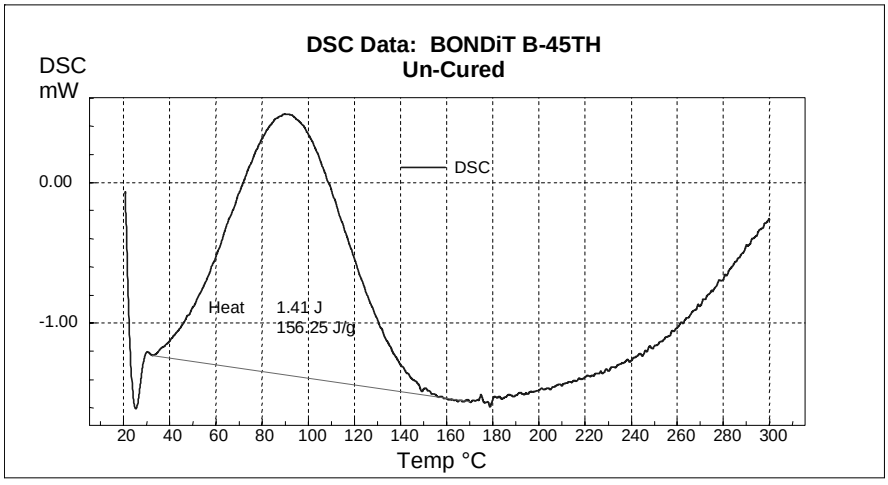


4. Degree of Cure Differential Scanning Calorimetry (DSC) was used for the cure study.

The adhesive was ***cured*** at typical (uncontrolled 16°C - 22°C) room temperature (RT) for 24 hours. It was observed in that period the surface remained somewhat tacky and the bulk of the material is very flexible.

An incomplete ***cure*** was suspected. To determine the cure schedule a DSC was performed on un-cured material from room temperature to 300°C at a rate of 5°C/min. Curing began at 30°C, peaked at about 90°C and ended at about 170°C. Degradation occurred at temperatures greater than 170°C. Normalized with respect to the sample amount, the heat of reaction during cure was measured to be 156.25J/g, the area under bell curve.

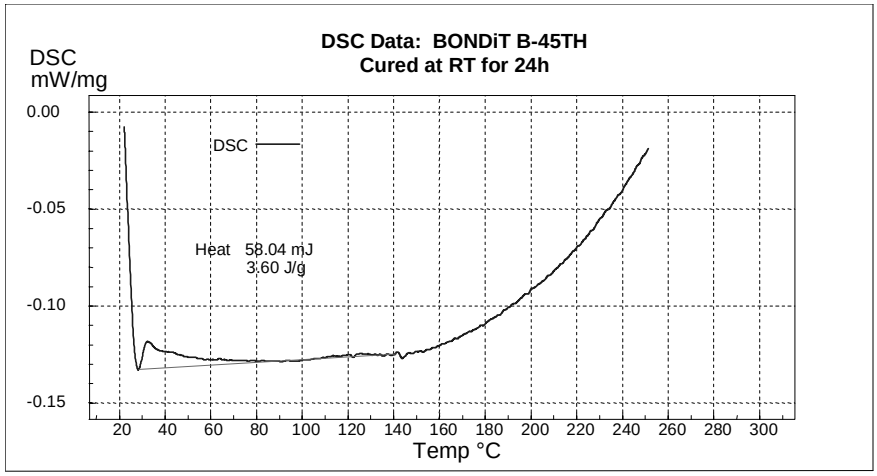
Figure 5. DSC Uncured B-45TH



A DSC scan was performed on a sample ***cured*** at room temperature (controlled 21°C) for 24 hours to measure the amount of cure. Normalized to sample weight yielded a heat of reaction of 3.60J/g thus making the sample 97.7% cured, meaning that a 24-hour, 21°C room temperature ***cure*** is sufficient for many applications. It is to be expected that a three (3) day ***cure*** or longer will produce further ***cure*** and improvement in physical properties for ambient cure applications.

$$\frac{156.25\text{J/g} - 3.60\text{J/g}}{156.25\text{J/g}} \times 100 = 97.7\%\ \textbf{cure}$$

Figure 6. DSC Cured B-45TH



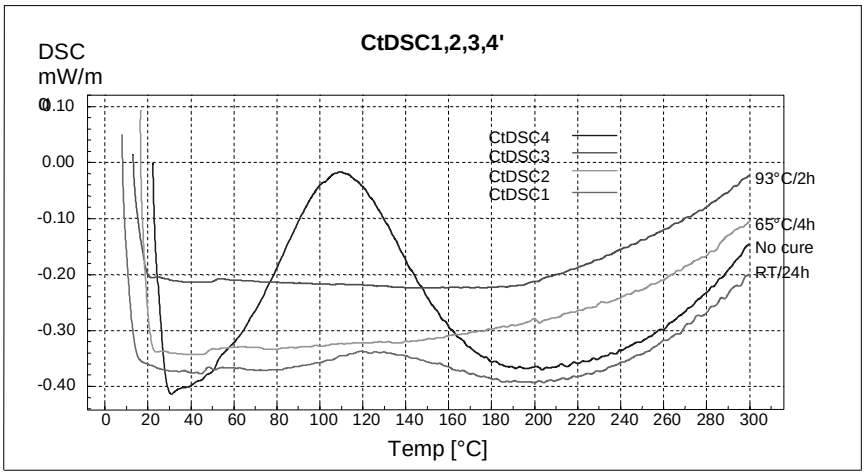
The extent of completion of the ***curing*** reaction can be easily evaluated by looking at the heat released when the epoxy is put through a thermal cycle. Figure 7 shows the profiles of the exothermic ***curing*** reaction for several ***curing*** methods. Table 3 lists the specific heat for three ***curing*** methods and an uncured sample.

Table 3. Specific heats of cured epoxy and uncured epoxy

Test #	Curing	Specific Heat of Curing reaction
CtDSC1	RT>24hrs	18.99 J/g (uncontrolled RT / 88.5% cure ref to CtDSC4 sample)
CtDSC2	65°C for 4 hrs	1.32 J/g
CtDSC3	93°C for 2 hrs	1.81 J/g
CtDSC4	no cure	165.25 J/g

From the specific heat data, it can be seen that optimized ***curing*** is correlate with ***curing*** temperature. For instance, at ambient temperature the material will never fully ***cure***, yet be sufficient for the application. Further studies by tensile strength tests indicate optimal operational ***cure*** is approximately 100°C for three (3) hours.
Figure 7 Extent of completion of curing as a function of cure method

Figure 7. Extent of completion of curing as a function of cure method



5. Environmental Test

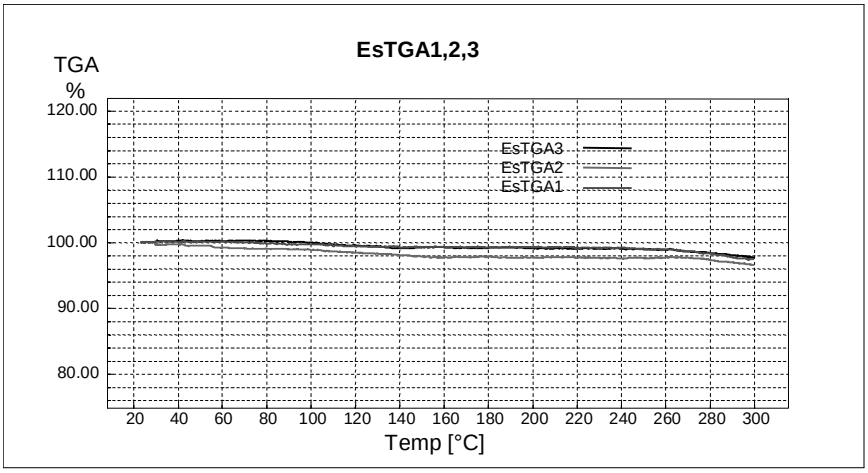
The three curing recommended curing methods were subjected to an ***environmental test***. The test was an 85RH / 85°C for duration of approximately four days. Figure 8 shows the weight loss of the tests processed using the three curing methods. Table 4 lists the percent weight loss of each of the tests.

Table 4. Percent weight loss of three curing methods after a 85/ 85 humidity test

Test #	Curing at 100°C	% wt. Loss
EsTGA1	24 hrs at RT (uncontrolled)	.343
EsTGA2	4 hrs at 65°C	1.176
EsTGA3	2 hr at 93°C	.286

The percent weight loss by EsTGA2 is the most significant. The weight lost by EsTGA1 and EsTGA3 are very similar. This result was not expected because of their very different curing methods. However, the RT was uncontrolled and over all the difference between the samples is relatively small. A larger statistical sampling would clarify the differences, if any.

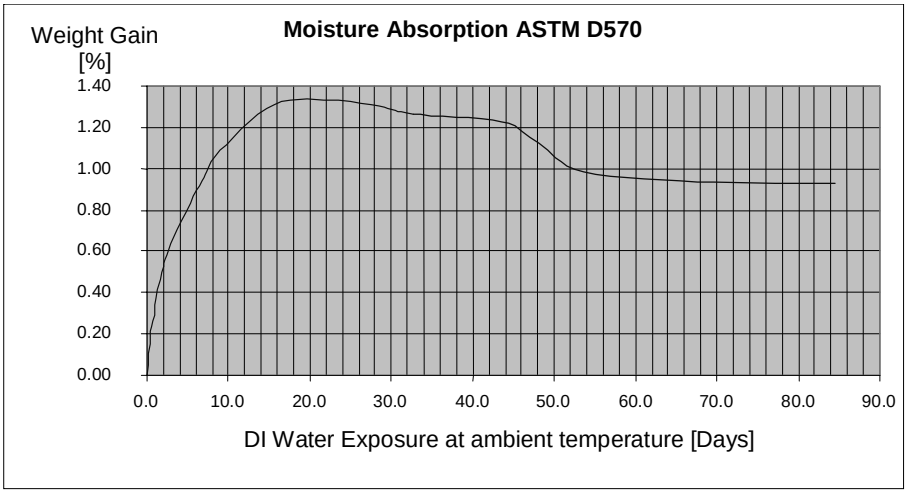
Figure 8. Weight lost for test runs after 4 days in an 85/85 chamber



6. Moisture Absorption

A moisture absorption test in DI water was conducted according to ASTM D570, showing an ultimate absorption of under 1% in equilibrium state after 51 days at uncontrolled lab room temperature (68-72°F days, 60-65°F nights).

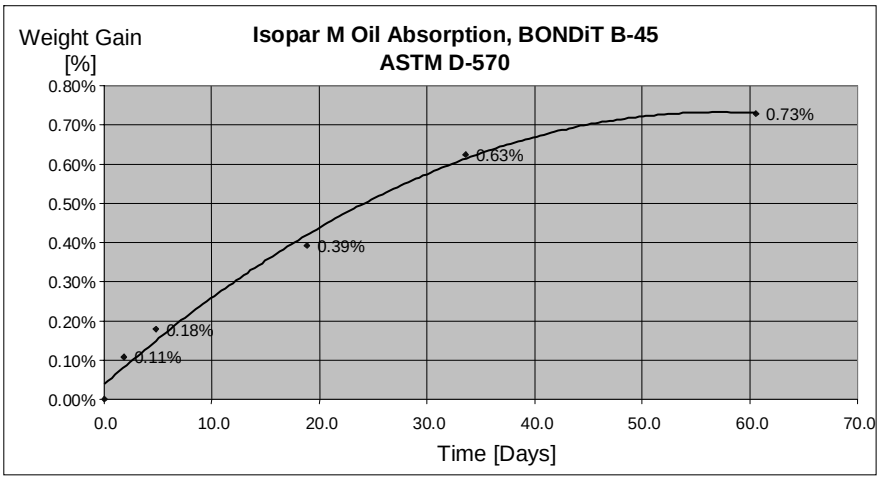
Figure 9. Moisture Absoprtion



7. Oil Absorption

Hand mixed samples were for oil absorption per ASTM D570 were also tested using Shell Oil Isopar M, resulting in an ultimate equilibrium of 0.73% in 60 days exposure.

Figure 10. Oil Absoprtion



8. Contribution of Solids Content

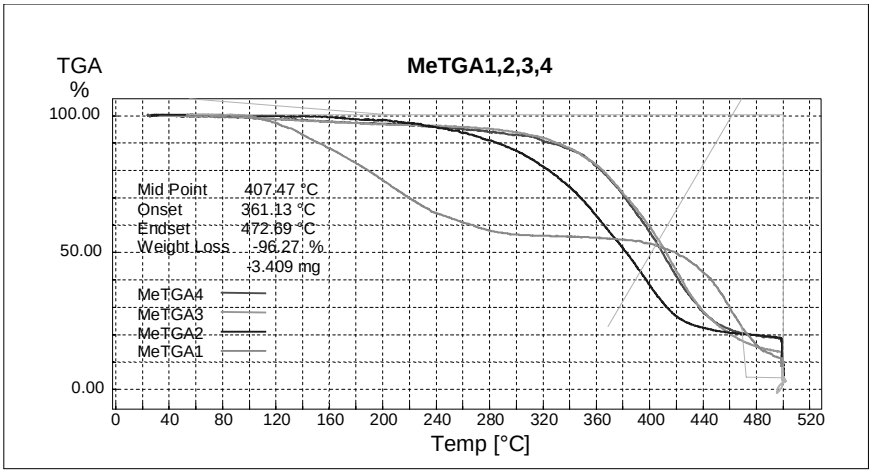
The total solids content contribution from the resin and the hardener is very comparable. Figure 11 shows the graphs of the total mass lost by each of the tests. A direct comparison of the total solids content in the epoxy mixture, the resin and the hardener can be made by regarding Table 5.

Table 5. Percent weight loss of epoxy materials and epoxy heated to 500°C for ½-hour

Test #	Material	% Weight Loss
MeTGA1	hardener	96.272
MeTGA2	resin	93.39
MeTGA3	static mixed	96.413
Me TGA4	hand mixed	95.309

There is not a lot of filler in the B-45TH epoxy system. It is interesting to note that there seems to be no appreciable difference between the solids content of the hand mixed and the static mixed epoxy.

Figure 11. Pyrolization profiles of the resin, hardener, and epoxy

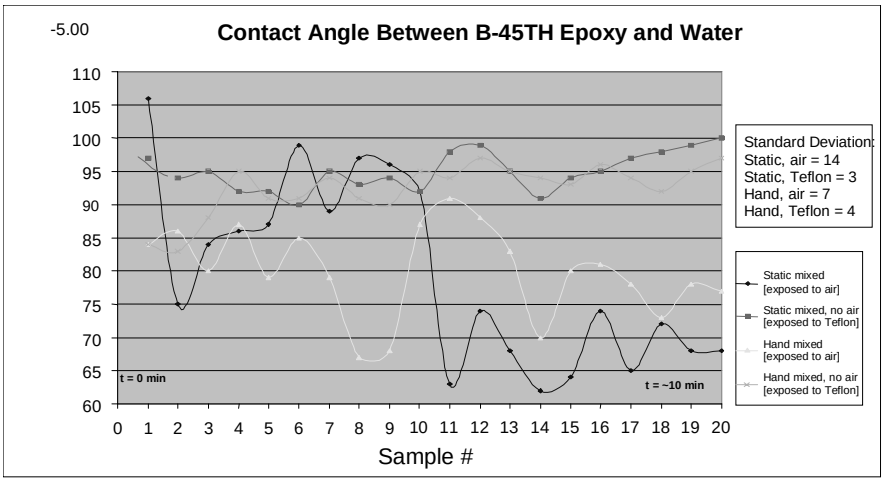


9. Surface Energy of Cured Material

The **surface energy** of B-45TH prepared by using a static mixer and by hand was looked at. The **surface energy** of the epoxy side exposed to air while curing and the side that was on the Teflon surface were compared. The data for the contact angle is shown in Figure 12.

The static mixed epoxy exposed to air while curing had an erratic contact angle profile. The standard deviation of the contact angle for the static, air set was approximately 14°. The backside of the epoxy had a much lower standard deviation of only 3°. In general, the backside that was not exposed to air during curing (static and hand mixed) had much higher contact angle values.

Figure 12. plot of the contact angle water on B-45TH



10. Mechanical Properties

Joint Strength

The data collected from the **joint strength** testing did not reveal any great disparity in strength between epoxy that is static mixed and hand mixed. Figure 13 shows the joint strength for 20 samples of the static mixed epoxy and the hand mixed epoxy.

Note the hand mixed sample had a higher standard deviation of .83 Kg compared to static mix of .64 Kg.

Figure 13. Joint strength of 20 samples of static and hand mixed epoxy

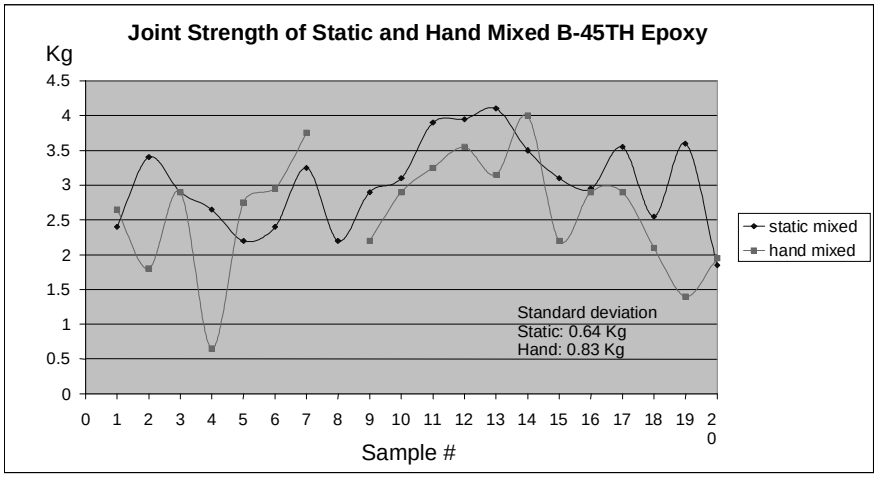


Table 6. Tensile Strength

B-45TH, 2 hrs set time, post Cured 2 hrs @ 100C
ASTM D638

Sample	Peak Stress PSI	Break Stress PSI	Break Elongation %	Yield Stress PSI	Yield Elongation %	Tangent Modulus PSI
Mean	1324.4	1264.8	103	1321.4	104.1	11932
Min	1262.1	1144.9	85.7	1262.1	84.7	10583
Max	1437.5	1422.1	130.4	1437.5	130	13510
Stdv	71.1	108.1	17	71.1	17.5	1133
%Cov	5.4	8.5	16.1	5.4	16.8	9
Medn	1293.7	1236	99.9	1293.7	97.4	11804

Figure 14.

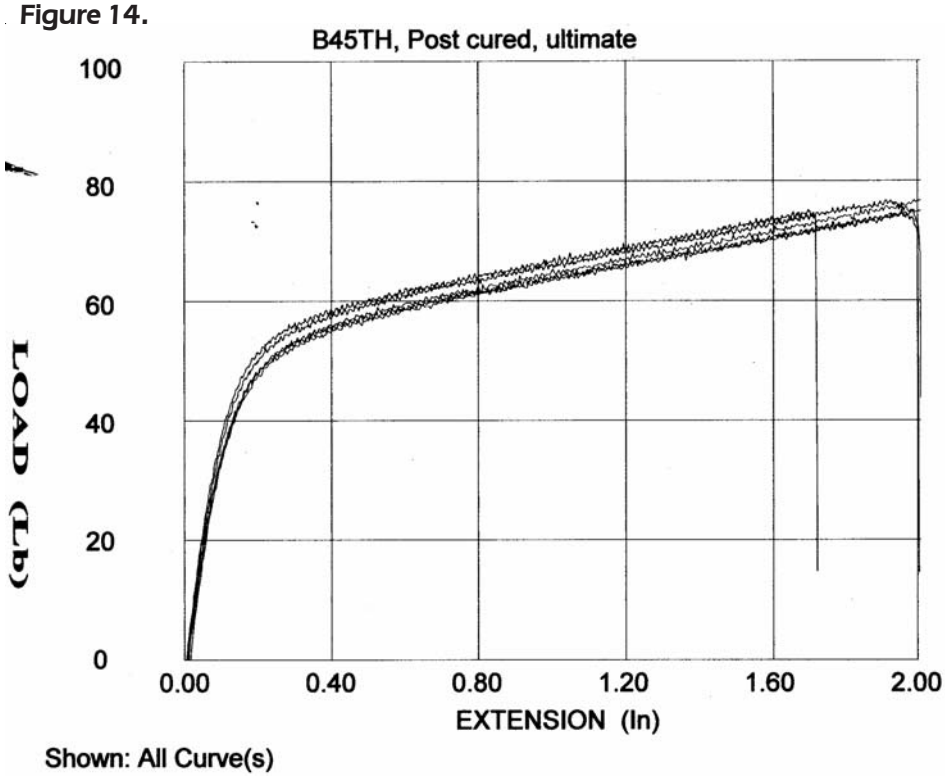


Table 7. Static Fatigue and Tear Strength

Fatigue and Tear Tests				
Fatigue tests		Static Load Lbs	Ultimate Tension PSI	Elongation %
150°F cure		46	835	215
Ambient	48	757	205	
Tear Strength Tear Die B		Static Load Lbs	Ultimate Tension PSI	Tear Strength lbs/in
150°F cure		28	310	121
Ambient		29	290	113

Table 8. Velocity of Sound, Density, Thermal Conductivit, Coefficicient of Thermal Expansion

Velocity of Longitudinal Sound Wave				Density	Thermal Conductivity	Coefficient of Thermal Expansion [Cte]
	Velocity	Temp	Frequency			From TMA, Fig 1
	m/sec	°C	KHz	g/cm^3	cal/(sec)(cm2) (°C)(cm)	ppm/°C
B-45TH	1923	25	200	1.046	300 x 10^{-6}	175
SeaWater	1531	25		1.025		
DI H2O	1496	25		0.998		
Dry Air	343.9	21		1.293		

Table 9. Lapshear Test per ASTM D3163
Lapshear test: ASTM D3163; all values mean.

ubstrate #1	Substrate #2	Cure Degrees F	Peak Stress PSI	Sample Range Hi/Lo Peak Stress PSI	Strain Peak Load %	Energy Peak Load In-Lb	Break Stress PSI	Elongation at Break %	Yield Stress PSI	Elongation at Yield %	Tangent Modulus PSI
HDPE	HDPE	150	295	299/290	1.65	2.96	290	1.7	295	1.72	2882
UHMW	UHMW	150	312	—	3.16	5.81	307	3.3	308	2.7	14184
UHMW	UHMW	150	279	325/256	2.22	3.69	279	2.4	274	2.3	14478
UHMW*	UHMW	Ambient*	283	308/246	2.2	3.69	282	2.2	279	2.1	15760
UHMW	Steel	150	424	484/364	1.74	4.69	424	1.6	424	1.6	33556
UHMW*	Steel	Ambient*	361	401/326	1.68	3.7	361	1.7	361	1.7	27291
PPS	SS	150	812	—	1.05	4.61	813	1.0	813	1.0	82478
PPS**	SS	Ambient**	492	—	0.77	2.25	490	0.8	492	0.8	77347
ABS	PVC	150	677	677/677	2.68	10.39	676	2.8	677	2.8	27247
PTFE***	PTFE	200	205	—	—	—	201	—	205	0.3	122693

* Ambient cure 31 days **Ambient cure 7 days ***Chemical etch surface treatment

Table 10. Peel Test per ASTM D638

Peel tests		
Substrate #1	Substrate #2	Failure mode
PBT	PET	cohesive/adhesive failure of PET film
PBT	Polyurethane foam	cohesive failure of PU foam
PEEK	PET	cohesive/adhesive failure of PET film
PEEK	Polyurethane foam	cohesive failure of PU foam
G10 fiberglass	PET	cohesive/adhesive failure of PET film
G10 fiberglass	Polyurethane foam	cohesive failure of PU foam
Neoprene	Steel	cohesive failure of rubber
Neoprene	Neoprene	cohesive failure of rubber
EPDM	Steel	cohesive failure of rubber

11. Bead Length from 50 ml Dual Cartiridge Through Static Mixer

Figure 14. Bead Lengths from 50 ml Cartridges of B-45TH Through Six Inch Static Mixer

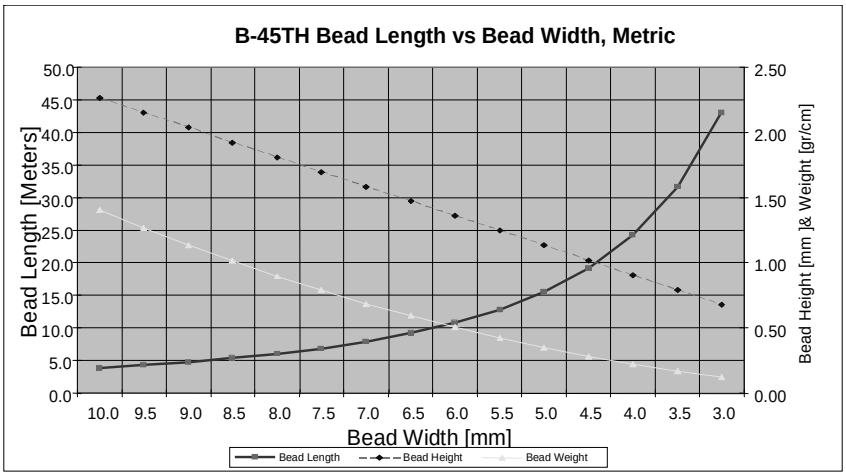


Figure 15. Bead Lengths from 50 ml Cartridges of B-45TH Through Six Inch Static Mixer