Product information

ANCAMIDE® 2424

Curing Agent

DESCRIPTION

Ancamide 2424 curing agent is a high performance, modified polyamide optimized for use with liquid epoxy resins in twocomponent structural adhesives. The product offers a number of advantages in ambient and heat-cured adhesives, particularly where rapid development of adhesive strength is of important. Ancamide 2424 combines the resiliency of traditional polyamides with state-of-the-art amine technology to produce a uniquely versatile curing agent for adhesive applications. Special features include increased reactivity versus conventional polyamides, enhanced low temperature cure, 100% solids and 3 hours thin film set at 77°F.

TYPICAL PROPERTIES

Property	Value	Unit	Method
Appearance	Clear amber liquid		
Color	9	Gardner	ASTM D 1544-80
Viscosity @ 77°F	14,000	cP	ASTM D-445-83, Brookfield, RVTD, Spindle 4
Amine Value	327	mg KOH/g	Perchloric Acid Titration
Specific Gravity			
@ 77°F	1.00		ASTM D 1475-85
@ 130°F	0.99		ASTM D 1475-85
Flash Point (closed cup)	274	°F	
Equivalent Wt/{H}	114		
Recommended Use Level	60	PHR, EEW=190	

ADVANTAGES

- Rapid development of adhesive strength due to increased reactivity
- Expanded working temperature range for 2K epoxies due to enhanced low temperature cure
- Excellent adhesion to metal and plastic substrates
- Reduced or eliminated need for costly accelerators
- Excellent water soak and environmental resistance
- DOT noncorrosive

APPLICATIONS

- Two-component epoxy adhesives for metal or plastic bonding
- Two-component structural adhesives where rapid development of handling strength is a key requiremen

STORAGE AND HANDLING

Refer to the Safety Data Sheet for Ancamide 2424 curing agent.

SHELF LIFE

At least 24 months from the date of manufacture in the original sealed container at ambient temperature. Store away from excessive heat and humidity in tightly closed containers.

TYPICAL HANDLING PROPERTIES

Property	Value	Unit	Method
Mixed Viscosity	20,000	cP	ASTM D-445-83, Brookfield, RVTD, Spindle 4
Gel Time5 (150 g mix @ 77°F)	60	min	Techne GT-4 Gelation Timer
Thin Film Set Time			
@ 77°F	3.0	h	BK Drying Recorder
@ 50°F	10	h	BK Drying Recorder
Peak Exotherm	285	°F	ASTM D 2471-71

* Ancamide 2424 curing agent formulated with standard Bisphenol-A based (DGEBA, EEW=190) epoxy resin.

SUPPLEMENTAL DATA

EXPERIMENTAL PROCEDURES:

To demonstrate its suitability for structural epoxy adhesives over a broad range of requirements, Ancamide 2424 curing agent was compared against Ancamide 350A curing agent accelerated with Ancamine K54, a package commonly used in adhesive applications. Toughened and nontoughened model formulations with 1:1 volume mix ratios were developed for each curing agent package and evaluated on multiple substrates under multiple cure schedules. The performance of ATBN-toughened adhesives was compared on galvanized and cold rolled steel after ambient cure, after ambient cure with a post bake, and after full heat cure. The performance of nontoughened adhesives based on each curing agent package was measured on cold rolled steel after each of the abovementioned cure schedules and on polycarbonate following ambient cure. Table 1 describes the composition of each model formulation, Table 2 summarizes the various cure schedules referenced throughout this bulletin and Table 3 specifies the metal substrates and test methods employed within this study.

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TABLE 1: MODEL ADHESIVE FORMULATIONS (1:1 BY VOLUME)

A Side	S1: 2424 Tough	S2: 350A/K54 Tough	S3: 2424 Nontough	S4: 350A/K54 Nontough	Method
DGEBA type liquid resin	64.0	64.0	64.0	64.0	
Microtuff 325F8	35.0	35.0	35.0	35.0	Microtuff 325F is manufactured by Barretts Minerals, Inc.
Cab-O-Sil TS-7209	1.0	1.0	1.0	1.0	Cab-O-Sil TS-720 is manufactured by Cabot Corporation
	100.0	**100.0**	**100.0**	**100.0**	
B Side					
Ancamide 2424	39.8		40.0		
Ancamide 350A		39.8		40.0	
Ancamine K54		2.9		3.0	
Epodil LV5 diluent	2.9	2.9	5.0	5.0	
ATBN 1300 X 16	21.4	21.4			Hycar ATBN 1300 X16 is manufactured by Goodrich Specialty
ATA-101	14.5	13.6	25.0	21.7	ATA-101 is manufactured by Alcan-Toyo America, Inc.
Microtuff 325	20.4	18.4	29.0	29.3	
Cab-O-Sil TS-720	1.0	1.0	1.0	1.0	
	100.0	**100.0**	**100.0**	**100.0**	

TABLE 2: CURE SCHEDULES

Schedule	Conditions
A	1 Day at 77°F
B	7 Days at 77°F
C	30 Days at 77°F
D	1 Day at 77°F + 30 min. at 250ºF
E	1 Day at 50°F
F	30 min. at 250ºF

TABLE 3: SUBSTRATES AND TEST METHODS

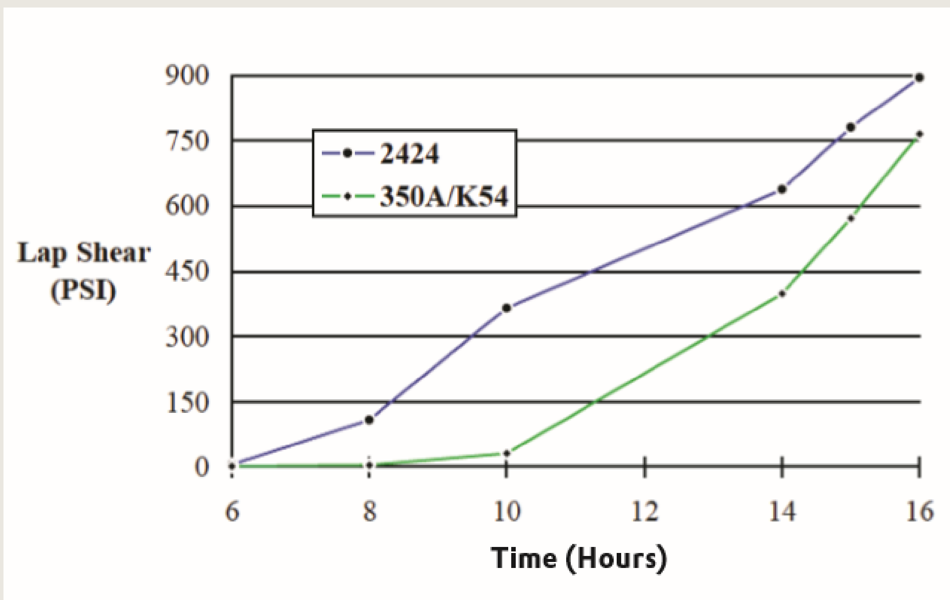
E60 EZG 60G	Galvanized Steel 0.030“ thick
B952 P60	Cold Rolled Steel 0.032“ thick
ASTM D 1002-72	Lap Shear Test Method
ASTM D 1876-72	T-Peel Test Method
ASTM B 117-85	Salt Spray Exposure Test Method
Lap Shear Sample Preparation and Testing	1“x4“ Cpupons
	0.5“ Bond Overlap
	0.020“ Bondline Thickness (controlled by glass beads)
	0.05“/ minute pull rate
T-Peel Sample Preparation and Testing	1“ x 4“ Coupons (90° bend at 1“)
	3“ Bond Overlap
	0.020“ Bondline Thickness (controlled by glass beads)
	10“/ minute crosshead speed
Sample Preparation	Acetone Wipe
Galvanized Steel	Dry Rag Wipe
Cold Rolled Steel	

RAPID DEVELOPMENT OF ADHESIVE STRENGTH:

End users of epoxy adhesives are interested in rapid development of adhesive strength without the brittleness and loss of robustness that often results when traditional systems are accelerated. Ancamide 2424 curing agent combines traditional polyamide technology, known for its robustness and suitability for a variety of performance requirements, with Evoniks' state-of-the-art amine technology to produce a versatile curing agent that provides superior development of adhesive strength versus traditional accelerated polyamide systems. The result is improved reactivity.

Figure 1 illustrates Ancamide 2424 curing agent's performance edge. Galvanized steel test coupons were bonded with toughened model adhesives S1 and S2 under ambient conditions, and the lap shear strength was measured as a function of time. As can be seen, Ancamide 2424 curing agent's ability to develop adhesive strength clearly surpassed the traditional system, developing over 100 psi in 8 hours and over 350 psi in 10 hours. In comparison, the traditional system barely began to react, achieving roughly 30 psi in 10 hours and essentially no strength in the time span of a standard 8-hour shift.

FIGURE 1: DEVELOPMENT OF LAP SHEAR STRENGTH AT 77°F



Substrate: galvanized Steel

EXPANDED OPERATING WINDOW FOR EPOXY SYSTEMS WITH IMPROVED LOW TEMPERATURE CURE:

Another weakness of traditional epoxy adhesives has been the inability to cure at lower temperatures. At 50°F and below, the reactivity of the amine groups in standard polyamides decreases dramatically. In fact, many will not cure at all at such temperatures. Ancamide 2424 curing agent has been engineered to provide enhanced reactivity to overcome the shortcomings, at low temperatures, of existing polyamide technology and to help expand the currently amenable operating window for epoxy systems. Figures 2 and 3 illustrate this point.

Figure 2 shows thin fi lm set time as a function of curing agent and temperature in both toughened and nontoughened adhesive formulations. A number of comparisons can be made that defi ne the Ancamide 2424 performance advantage. First, the relative values of thin fi lm set time favor Ancamide 2424 curing agent. In each formulation and under each cure condition, the Ancamide 2424-based formulation had shorter thin fi lm set time than the accelerated Ancamide 350A system, with a minimum of 3 hours at 77 °F. Second, the relative eff ects of lower temperature were less dramatic with Ancamide 2424 curing agent. Again in each case, the changes in thin fi lm set time at 50°F versus 77°F were smaller for the Ancamide 2424-based adhesives. Finally, including a rubber modifi er had less impact on the reactivity of the formulations containing Ancamide 2424. The increase in thin fi lm set time for both toughened and nontoughened Ancamide 2424 systems was 7 hours, while the thin fi lm set time of the toughened and nontoughened accelerated Ancamide 350A systems increased 12 and 9 hours, respectively. Not only was the impact on reactivity less pronounced with Ancamide 2424 curing agent, but it was not temperature dependent

FIGURE 2: THIN FILM SET AS A FUNCTION OF FORMULATION AND TEMPERATURE

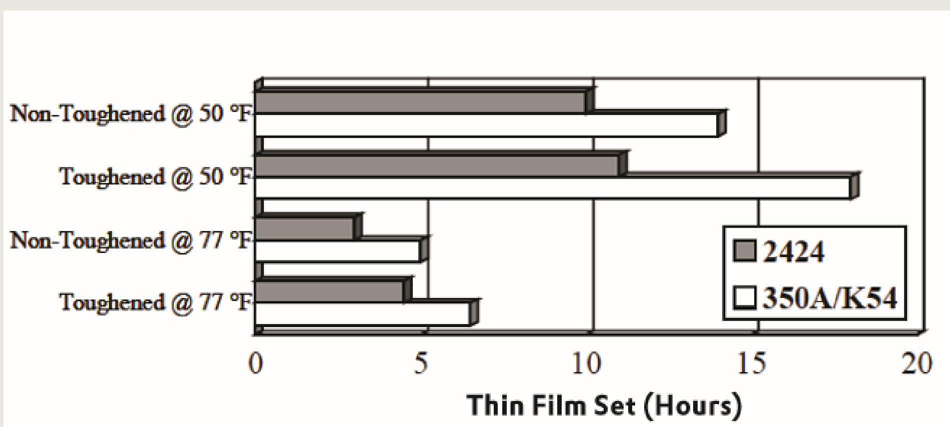


Figure 3 demonstrates the practical benefi ts of improved reactivity as indicated by the development of lap shear strength at 50°F. Whether toughened or nontoughened and whether bonding galvanized or cold rolled steel, model adhesives based on Ancamide 2424 curing agent provided superior lap shear strength aft er 24 hours at 50°F. These data parallel the results collected at 77°F and reinforce the practical benefi ts of working with a more reactive curing agent.

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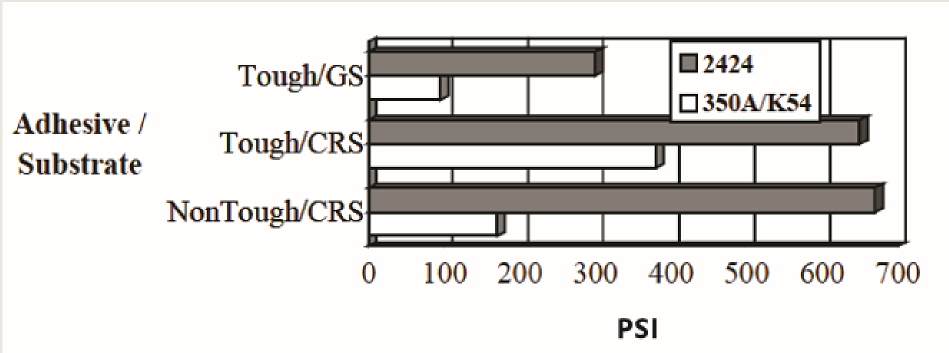
FIGURE 3: LAP SHEAR STRENGTH AFTER 24 HOURS AT 50°F



EXCELLENT ADHESION TO METAL AND PLASTIC SUBSTRATES:

To demonstrate utility across a broad range of applications, the model adhesives were used to bond a variety of substrates under a variety of cure schedules. As described above, the toughened systems (S1 and S2) were used to bond galvanized and cold rolled steel, and the nontoughened systems (S3 and S4) bonded cold rolled steel and polycarbonate. Lap shear and T-peel data were collected for the metal substrates, and lap shear data were collected for the polycarbonate bonding following an ambient temperature cure.

Figures 4, 5, 6 and 7 illustrate Ancamide 2424 curing agent's versatility beyond just rapid development of lap shear strength. The lap shear results were impressive, even aft er post-baking or full heat cure.

On galvanized steel, Ancamide 2424 consistently matched or exceeded the performance level of the traditional system and showed no loss in robustness or increase in brittleness as might be expected of a more reactive material.

Mode of failure is as equally important as the absolute level of lap shear strength. All systems that were tested on metal substrates provided cohesive failure, where the adhesive pulled apart but the bond at the substrate interface remained intact. In fact, the galvanized steel coupons used throughout the study were consistently stretched prior to failure.

Adhesive failure was observed on polycarbonate, which was not surprising given that polycarbonate resin provides a low energy surface and our model formulations were not optimized for plastic bonding. However, with model adhesive S3, we achieved 240 psi of lap shear strength on polycarbonate aft er one week at 77°F.

FIGURE 4: TOUGHENED LAP SHEAR ON GALVANIZED STEEL AS A FUNCTION OF CURE SCHEDULE

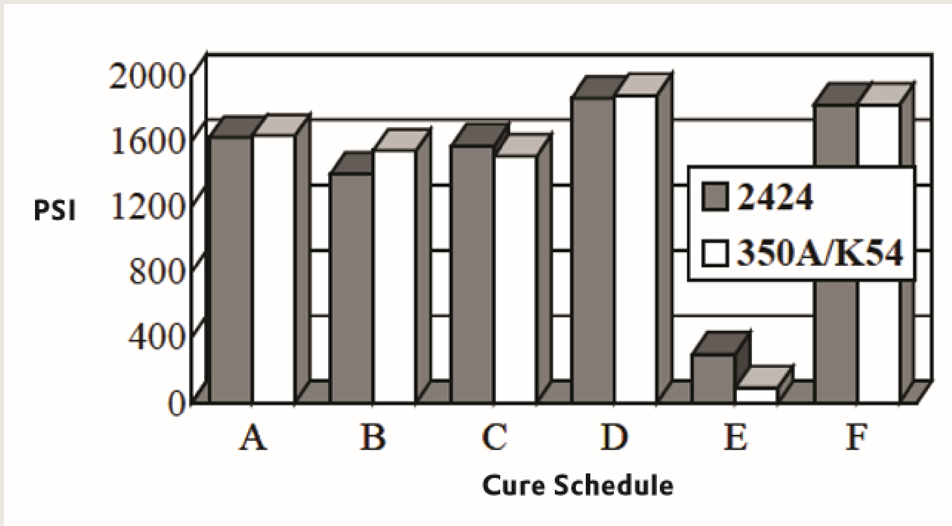


FIGURE 5: TOUGHENED LAP SHEAR ON COLD ROLLED STEEL AS A FUNCTION OF CURE SCHEDULE

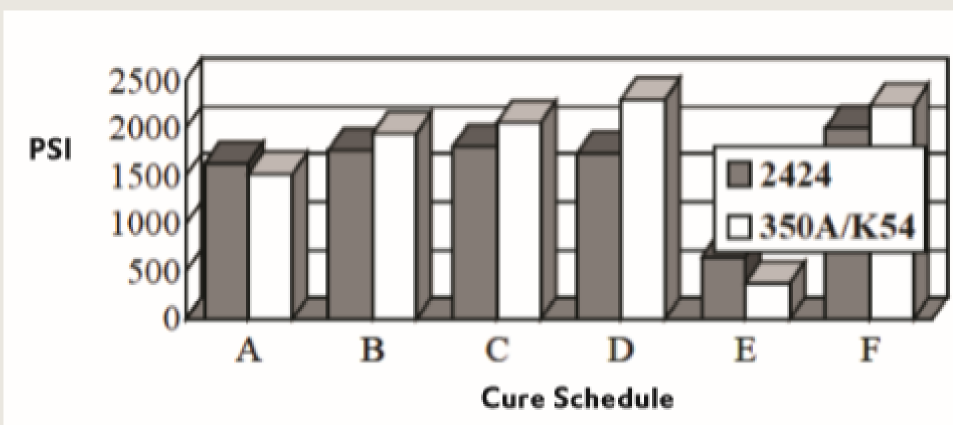
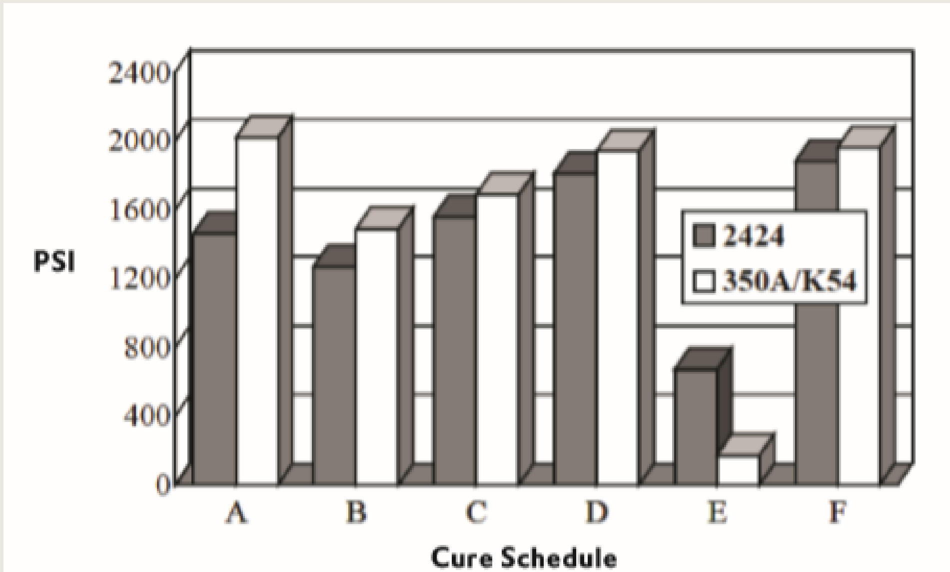


FIGURE 6: NONTOUGHENED LAP SHEAR ON COLD ROLLED STEEL AS A FUNCTION OF CURE SCHEDULE

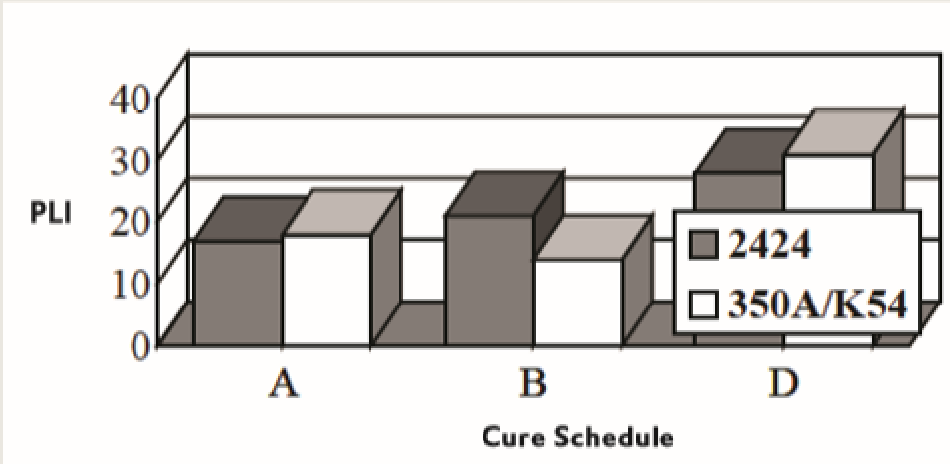


One must be careful when interpreting the nontoughened lap shear results. At fi rst glance it appears that the traditional polyamide adhesive off ers a signifi cant advantage over Ancamide 2424 curing agent aft er 24 hours at 77°F (see cure schedule A). However, the lap shear strength values decreased considerably over the next week or so, as evidenced by the cure schedule B value. This apparent high level of strength followed by relatively weaker lap shear values was caused by incomplete cure in the traditional system. As a partially cross-linked network, the traditional polyamide exhibited a pseudo toughness that is not indicative of its ultimate properties. As a more reactive curing agent, Ancamide 2424 curing agent was less susceptible to this phenomenon. Therefore, what appeared at fi rst to be a shortcoming of Ancamide 2424 was actually indicative of its higher reactivity and the degree of cure attainable in a 24 hour period.

Peel strength for the toughened adhesives (S1 and S2) was evaluated on cold rolled steel aft er three diff erent cure schedules (A, B and D). Ancamide 2424 curing agent’s performance paralleled the traditional polyamide system, consistently gaining strength with a higher degree of cure. As with the lap shear testing, the mode of failure was cohesive with all samples.

The pseudo toughness of the traditional polyamide system is evident in the peel strength value for adhesive S2 aft er 7 days at 77°F. S2 achieved 18 pli of peel strength aft er 1 day but only 14 pli aft er 7 days. Ancamide 2424 curing agent achieved 17 pli and 21 pli under the same conditions, respectively.

FIGURE 7: TOUGHENED LAP SHEAR ON GALVANIZED STEEL AS A FUNCTION OF CURE SCHEDULE



ADHESIVE INTEGRITY AFTER EXPOSURE TO HOT WATER AND SALT FOG:

Reliability is a primary concern of the adhesive end user. To gauge an adhesive's ability to maintain bondline integrity and adhesive strength, lap shear was measured for the toughened systems (S1 and S2) aft er prolonged exposure to hot water and salt fog. In the so-called hot wet test, bonded samples were submerged in water for 7 days at 160°F. Salt fog exposure was 30 days in a 5% NaCl fog at 95°F.

The results are illustrated in Figures 8 and 9. Figure 8 shows the eff ects of exposure following a one week ambient temperature cure. Figure 9 shows the eff ects on samples cured for 30 minutes at 250°F. In both sets of tests, Ancamide 2424 curing agent matched the performance of the traditional polyamide and further demonstrated its suitability for use in structural epoxy adhesive applications.

FIGURE 8: LAP SHEAR STRENGTH AFTER EXPOSURE CURE SCHEDULE B

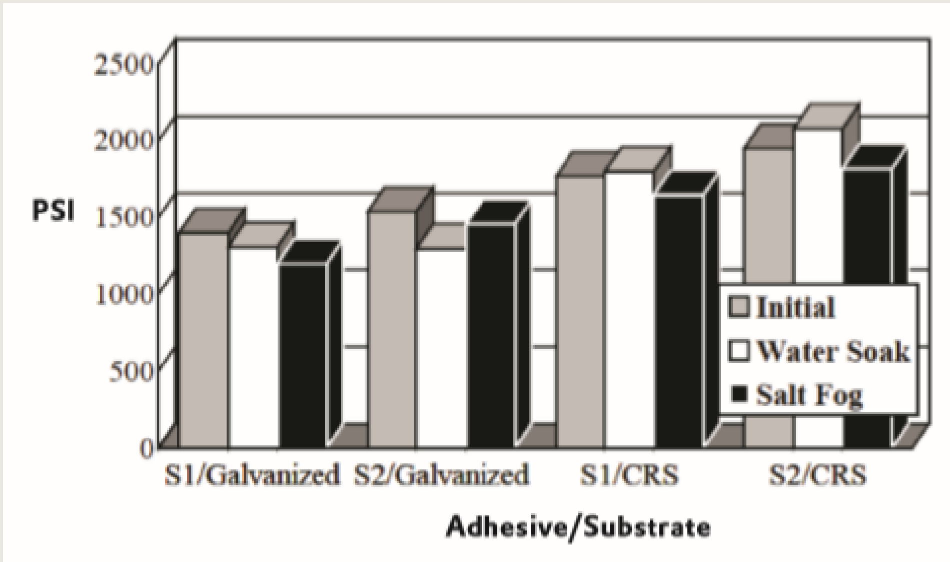
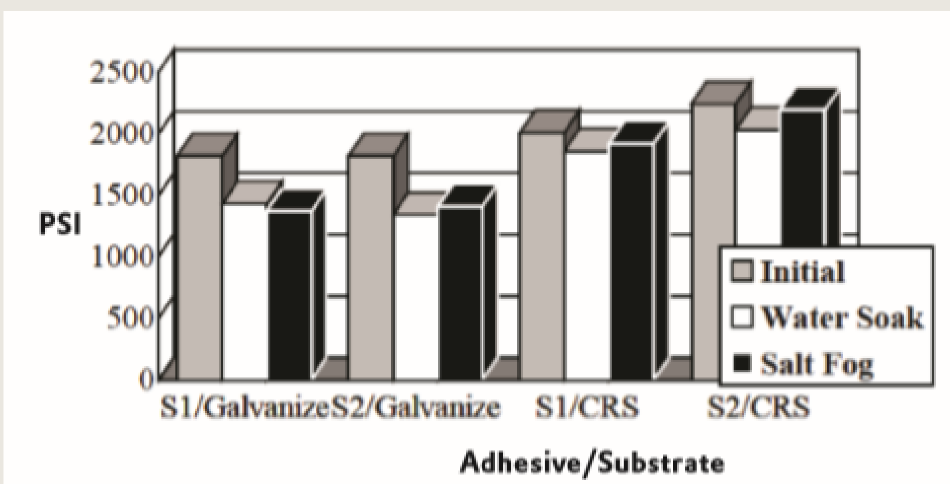


FIGURE 9: LAP SHEAR STRENGTH AFTER EXPOSURE CURE SCHEDULE F



APPENDIX 1: EXPERIMENTAL RESULTS

Figure 1	**Lap Shear (PSI)**	
Time (Hours)	**2424**	**350A / K54**
6	6	2
8	109	4
10	365	32
14	638	398
16	895	766
Figure 2	**Thin Film Set (Hours)**	
System / Temperature	**2424**	**350A / K54**
Nontough / 50 °F	10	14
Tough / 50 °F	11	18
Nontough / 77 °F	3	5
Tough / 77 °F	4.5	6.5
Figure 3	**Lap Shear (PSI)**	
Adhesive / Substrate	**2424**	**350A / K54**
Toughened / Galvanized Steel	300	95
Toughened / Cold Rolled Steel	650	380
Nontoughened / Cold Rolled Steel	670	170
Figure 4	**Lap Shear (PSI)**	**Toughened on Galvanized**
Cure Schedule	**2424**	**350A / K54**
A	1620	1630
B	1400	1540
C	1570	1510
D	1860	1880
E	300	95
F	1820	1820
Figure 5	**Lap Shear (PSI)**	**Toughened on Galvanized**
Cure Schedule	**2424**	**350A / K54**
A	1630	1520
B	1770	1950
C	1810	2060
D	1740	2300
E	650	380
F	2010	2240

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APPENDIX 1: EXPERIMENTAL RESULTS (continued)

Figure 6	Lap Shear (PSI)	Nontoughened on Cold Rolled	
Cure Schedule	**2424**	**350A / K54**	
A	1460	2020	
B	1270	1490	
C	1270	1690	
D	1810	1940	
E	670	170	
F	1880	1960	
Figure 7	**T Peel (PLI)**	**Toughened on Cold Rolled**	
Cure Schedule	**2424**	**350A / K54**	
A	17	18	
B	21	14	
D	28	31	
Figure 8	**Lap Shear (PSI)**		
Adhesive / Substrate	**Initial**	**Water Soak**	**Salt Fog**
S1 / Galvanized	1400	1310	1200
S2 / Galvanized	1540	1300	1460
S1 / Cold Rolled	1770	1800	1650
S2 / Cold Rolled	1950	2080	1820
Figure 9	**Lap Shear (PSI)**		
Adhesive / Substrate	**Initial**	**Water Soak**	**Salt Fog**
S1 / Galvanized	1820	1430	1370
S2 / Galvanized	1820	1340	1410
S1 / Cold Rolled	2010	1860	1920
S2 / Cold Rolled	2240	2030	2200

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EVONIK OPERATIONS GMBH
Business Line Crosslinkers
Paul-Baumann-Str. 1
45764 Marl
Germany

www.evonik.com/crosslinkers
Product Information: APCSE@evonik.com
Sample Request: APCSE@evonik.com

EVONIK CORPORATION
Business Line Crosslinkers
7001 Hamilton Boulevard
Trexlertown, PA 18087
USA

CrosslinkersProdinfo@evonik.com
Crosslinkers-Samples@evonik.com

EVONIK SPECIALTY CHEMICALS (SHANGHAI) CO., LTD.
Business Line Crosslinkers
55, Chundong Road
Xinzhuang Industry Park
Shanghai, 201108
China
CL-Asiainfo@evonik.com
CL-Asiainfo@evonik.com

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