TEXAS RESEARCH INSTITUTE AUSTIN, INC.
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Introduction of **TRI-SealPlus** Seal Coat for ***BondCoat*** ä System Accelerated Life Test Results

As reported in early 1999, the impending discontinuation of the current Metco AP led to the development by Texas Research Institute Austin, Inc. (TRI/Austin) of a replacement seal coat for our BondCoat system. The new seal coat, **TRI-SealPlus**, was developed to outperform Metco AP by providing better sealing capabilities of the plasma-sprayed BondCoat. Results of accelerated life testing (ALT), using two popular molding compounds and adhesive systems, indicates that **TRI-SealPlus** can be expected to perform exceptionally well in a cathodic marine environment.

Highlights of the new **TRI-SealPlus**:

- Has higher percent solids content than the Metco AP coating and less Volatile Organic Compounds (VOCs).
- Passed the water drop test developed by NUWC (electrical probe used to apply 500 Vdc from a megohmmeter to water pooled on surface of coated substrate).
- Offers enhanced adhesive bond strength reliability.
- ALT results to 144 days show excellent bond strength durability, greater than 60 pounds per inch width (piw) for one of the molding systems and nominally 20 piw for the other. The best standard rubber-to-metal bond systems failed entirely within 30 days.
- The adhesive bond durability results with TRI-SealPlus was significantly better in this test than were the results achieved with the Metco AP seal coat during the initial project efforts to develop BondCoat.
- Will be commercially available through TRI without risk of discontinuation or modification.

Investigation of Candidate Replacement Seal Coat Systems

A variety of candidate seal coat systems were evaluated before we developed **TRI-SealPlus**. No commercially available system offered all of the required properties: high solids content, long-term bondability in a cathodic saltwater environment, ease of application, environmentally friendly applications, guaranteed availability, and cost and quality control.

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Accelerated Life Testing

Four 9 x 9 x 0.25-inch 316L stainless steel plates were used in this test: two were treated with BondCoat and two were left bare and only grit blasted with aluminum oxide prior to cleaning, priming and overmolding. The test configuration of the four plates is shown in Table 1. The bare steel plates were cleaned with methyl propyl ketone (MPK) after surface abrasion. The BondCoat plates were also cleaned with MPK prior to application of the TRI-SealPlus. The primers and molding materials used were: PRC-DeSoto (formerly Courtaulds Aerospace) Corporation's PR-421 (the replacement for the standard, but discontinued, PR-420) and PR-1547 polyurethane molding compound; and Conap Corporation's AD-1146 primer and EN-1156 polyurethane molding compound.

After priming the plates, molding compound was applied in a layer approximately 0.25 ± 0.02 inches thick and formed in isolated strips, referred to as coupons, 0.75 inches wide. Application and curing of primers and molding compounds was conducted in accordance with the manufacturers' recommendations. Figure 1 shows one of the BondCoat test plates.

Table 1. Test Plate ALT Test Article Configuration

Plate Number	Substrate	Seal Coat	Primer	Molding Compound
1	Stainless Steel	None	PR 421	PR 1547
2	BondCoat Plasma Coating	TRI-SealPlus	PR 421	PR 1547
3	Stainless Steel	None	AD 1146	EN1556
4	BondCoat Plasma Coating	TRI-SealPlus	AD 1146	EN 1556

After molding, baseline peel tests were performed. Electrode-grade zinc anodes were attached to the test plates during immersion in a 70°C saltwater bath. Test conditions included continuous purging with air (pumped through "bubbling" stones) and stirring to maintain a realistic dissolved oxygen content (which cannot be maintained at elevated temperatures without this type of intervention). The plates were periodically removed from the bath to conduct peel tests in an Instron universal testing machine. Table 2 shows the time of each test and the peel strength value for each coupon peeled from its test plate. Figure 2 shows the tabulated data over the 144-day test period.

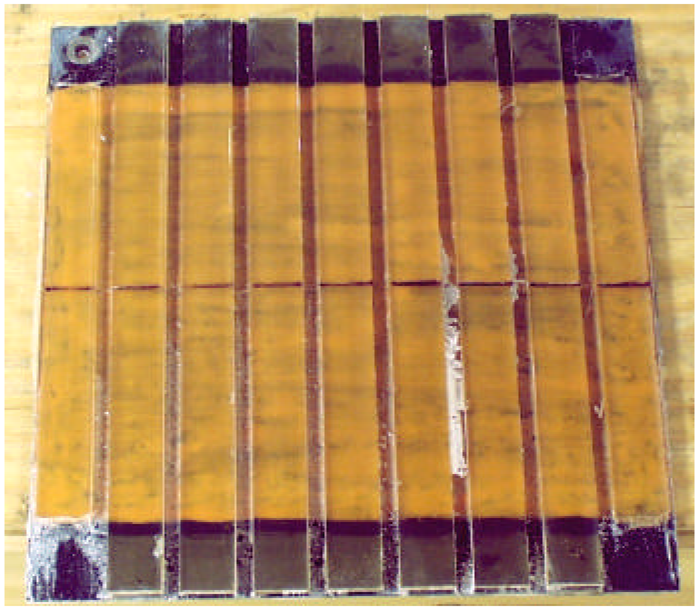

Figure 1. Stainless steel test plate with isolated peel coupons of PR-1547 molding compound, PR-421 primer, TRI-SealCoat and BondCoat.

Table 2. ALT Peel Test Data

Exposure (days)	**Plate 1** SS 421/1547 (piw)	**Plate 2** BC/SP 421/1547 (piw)	**Plate 3** SS 1146/1556 (piw)	**Plate 4** BC/SP 1146/1556 (piw)
3	63	60	1	59
10	35	62	0	53
20	11	69		71
30	0	72		73
42		64		97
76		45		84
111		29		73
144		19		64

Notes: SS = 316L; BC/SP = BondCoat with TRI-SealPlus; 421/1547 = PRC-Desoto Molding System; 1146/1556 = Conap's Molding System; piw = Pounds per Inch Width

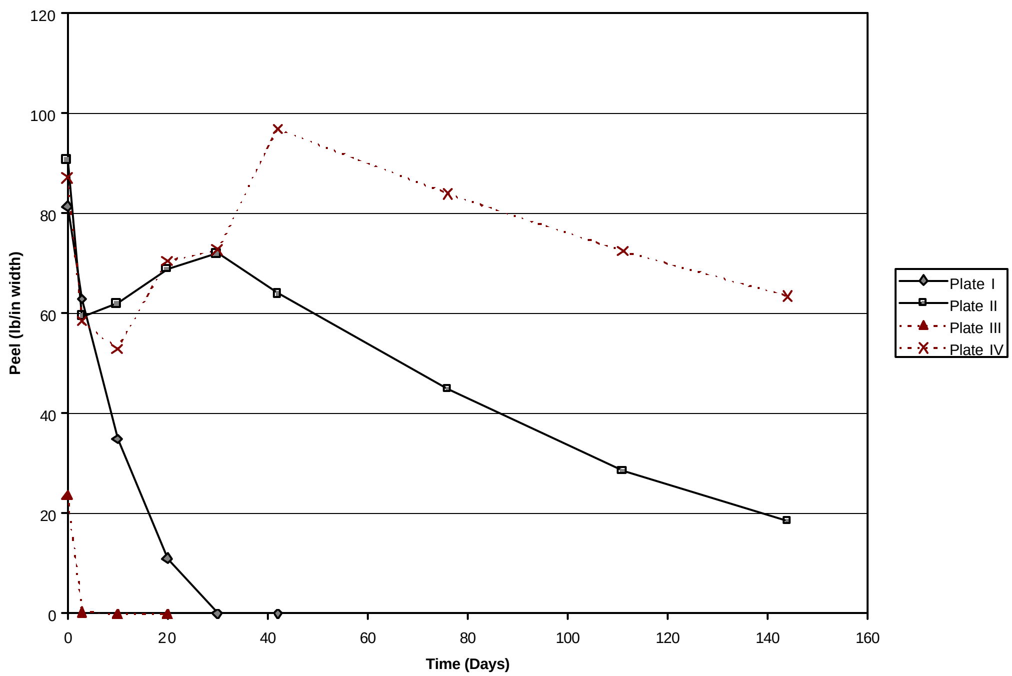

Figure 2. ALT Peel Test Data to Test Completion of 144-days

From the data shown, the Conap system performed very poorly on bare, cathodically polarized stainless steel in an aggressive, aerated, saltwater environment. The PRC-Desoto PR-421 and PR-1547 system did marginally better but, still showed rapid adhesive bond degradation within 30 days. The plates treated with BondCoat and TRI-SealPlus continue to show excellent results after 42 days for both priming and molding systems. In this test, BondCoat has added approximately ten times to the life of the Conap system and doubled the life of the PRC-Desoto system.

TRI-SealPlus versus Metco AP Comparison

During the initial project development of BondCoat, ALT results with coupons made from the PR-1547 and PR-420 molding system applied over Metco AP seal coat on stainless steel were good, but not as good as those for the same molding compounds applied to TRI-SealPlus. In the first test, bond strength degraded to below 40 piw after 30 days of exposure. Thise is actually a very respectable bond strength; however, Figure 2 shows that the same configuration with TRI-SealPlus dropped below 40 pli after 80 days of exposure - an **increase in bond life of over 2.5 times**.

A discussion of ALT acceleration rates as a function of service temperature exposures

TRI/Austin developed mission profiles for Navy submarines under contracts with the Naval Research Laboratory - Underwater Sound Reference Detachment (NRL-USRD) during the Sonar Transducer Reliability Improvement Program (STRIP).

Accelerated life testing plans were based on analyses of the various temperature ranges and means for all phases of outboard hardware life. These included transportation and storage, installation and maintenance, and service periods. These mean temperature and duration inputs are necessary in order to scientifically estimate the degree of accelerated aging that can be achieved for given laboratory temperature exposures. ***[Note: This discussion basically applies to the single exposure to saltwater soak and does not include other environments included in a comprehensive ALT, i.e., in-air, hydrostatic pressure, wet/dry thermal cycling, etc.]***

Determining the average yearly temperature uses the following calculation:

$$\bar{T} = \frac{\sum T_n t_n}{8766}$$

T = temperature in Kelvin per step
t = time step in hours
n = the total number of time steps

Also, the following must be true:

$$\sum t_n = 8766 \text{ hours.}$$

Various environmental service temperature calculations will be discussed: 10, 16, 20, 25 and 30°C. Using TRI/Austin-developed ALT software with an activation energy of 13 kcal/mole K, the number of laboratory test hours to equal one equivalent year (EY) of service were calculated for each average yearly temperature mean. Note that one average yearly temperature is used at a time in this discussion; during an ALT of a particular part or system, multiple temperatures and their duration (in hours) are used to better describe the average exposure temperature over the course of a year to represent the variable environmental conditions.

The 13 kcal/mole K activation energy was agreed to by STRIP investigators as reasonable based on a best fit estimation of diffusion of water in polymer materials common to outboard sonar transducers, cables and connectors.

Table 3. Mean Service Temperature and Calculated Time to Achieve One EY with Laboratory Exposure of 70°C

Mean Temperature (Service)	**Lab Hrs per EY**	**EY per Lab Year**	**Total EY @ 144 days** (3456 hrs)
10	118	74	29
16	197	45	17
20	273	32	12
25	407	22	8
30	600	15	6

As noted previously, these calculations are based solely on acceleration factors related to the 13 kcal/mole activation energy and the difference between mean service temperature and the laboratory exposure temperature. In a comprehensive ALT test, designed to emulate the mission profile for a transducer or a cable connector, the overall acceleration rates tend to be much lower since some exposures cannot be accelerated at these rates without using damaging (and unrealistic) laboratory exposures. This would apply to such things as hydrostatic pressure testing at ambient temperature, a very necessary test exposure, but not one that carries a high acceleration factor. The in-air exposures are also less efficient since the service mean temperatures, especially for maintenance (docking) periods, are relatively high compared to in-water service and therefore accelerated less in a 70°C exposure.

Figures 3, 4 and 5 show the coupon ALT peel data graphed to reflect EYs of aging for mean service temperatures of 10, 20 and 30°C. They readily show the relationship between service and laboratory temperature exposures for interpreting actually aging rates. It should be pointed out that the primary purpose of the coupon ALT was to compare the baseline rubber to metal bond system to the BondCoat with TRI-SealPlus using two popular molding/adhesive systems. We do not purport herein to state how much aging the test represents for submarine installed connectors, but an estimate of between 7 to 12 years is reasonable.

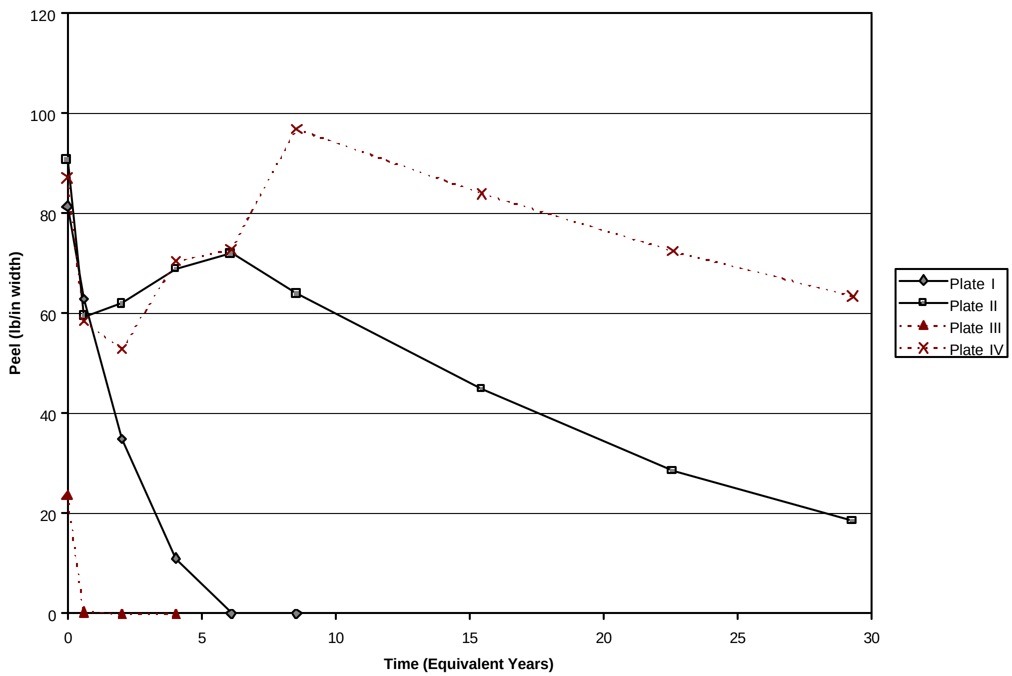

Figure 3. 10°C Average Temperature Exposure Graph

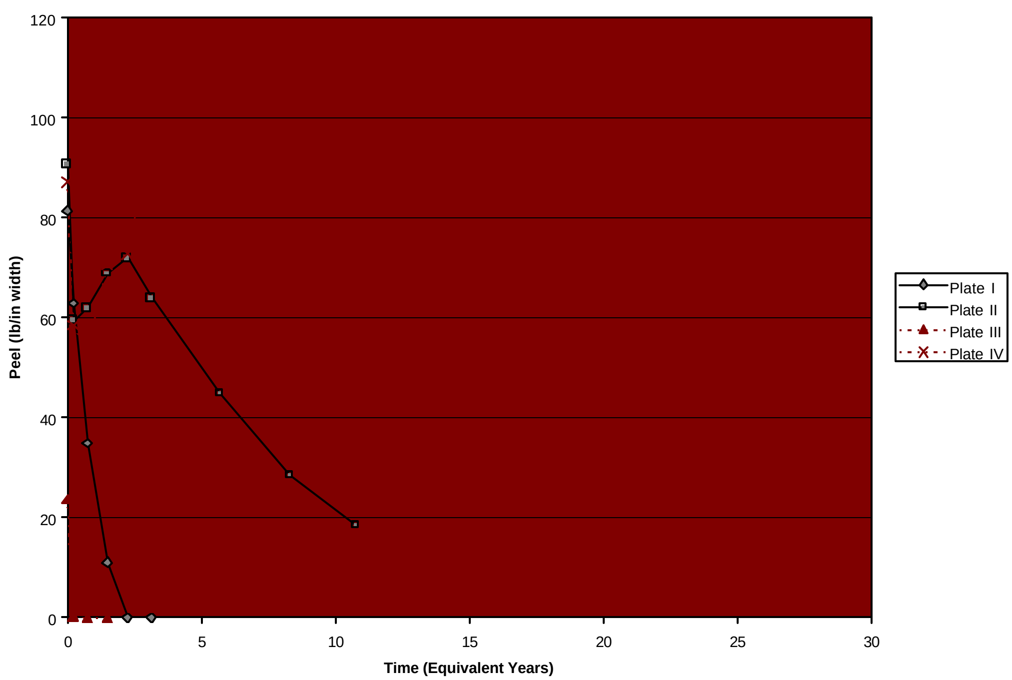

Figure 4. 20°C Average Temperature Exposure Graph

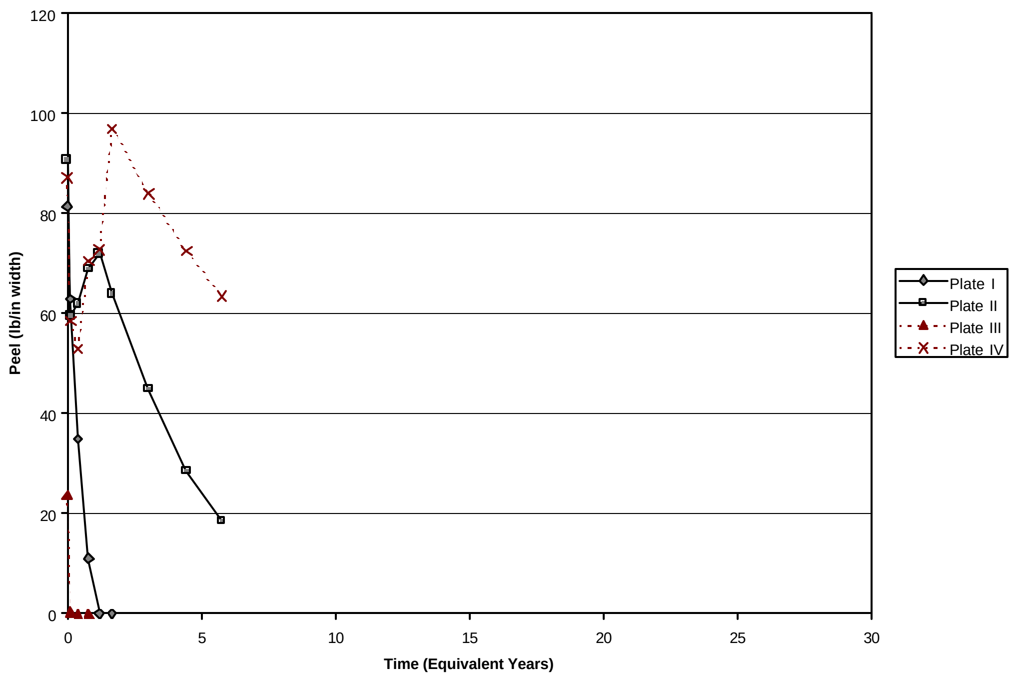

Figure 5. 30°C Average Temperature Exposure Graph

Conclusions

TRI-SealPlus improved bond life over the current Metco AP product (~2.5 times) and the standard rubber to metal connector molding (~5 times) in this ALT. With its higher percent solids and proven adhesive bondability, TRI-SealPlus appears to be a suitable replacement for the discontinued Metco AP in applications with BondCoat.

Temperature and other environmental factors will affect the performance of any adhesive bond system. Actual life expectancy is difficult to predict without extensive laboratory testing; however, the BondCoat/SealCoat combination clearly indicates stronger, longer-lived adhesive bonds over standard configurations in a cathodic marine environment.