Accelerated Acid-Etch Test at Solutia

gradient-oven, a unique lab oven simulating multiple temperatures on one test panel

Acid etch, or environmental etch, is a defect that can occur in any exterior paint finish. However, it is a major concern for the automotive industry. The result of acid etch may appear as rings on a car's finish. These rings may seem like water spots, but in actuality, damage has been done to the finish, so washing does not remove the rings. Certain geographical areas are affected more than others – areas adjacent to heavily industrialized cities usually have high incidents of acid etch complaints.

Historically, isocyanate-crosslinked coatings outperformed melamine-crosslinked coatings for acid-etch resistance. However, melamine-crosslinked coatings are still in use because they are more cost effective and have better mar resistance.

The automotive industry has primarily studied and tried to eliminate the paint defect caused by acid etch. An accelerated acid etch test using the BYK-Gardner gradient-oven facilitates evaluation of different coating systems.

This relatively simple test yields a pair of values, the minimum spot temperature (MST) and the scaled acid value. These values are used to rank the severity of the acid damage to a coating. To make the comparison of coatings in this brief study easier, one acrylic polyol and either an isocyanate resin or a melamine resin were used as the crosslinker.

Procedure

- Prepare a panel for testing by coating a film on a 4" x 22.5" x 0.032" steel (polished) panel using the formulation and cure parameters for the specific material. Sometimes a post-cure time period is recommended before the acid-etch test is run.
- Set the gradient-oven for a 30-minute time cycle using a temperature range of 35 - 75 °C as a continuous gradient. The oven has 45 individual temperature zones.
- Label the panels with the supplied paper strips which are numbered from 1 - 45 corresponding to the 45 gradient temperature zones. Directly opposite each number on the strip, use a pipette to dispense approximately two drops (0.4 grams) of a 10% sulfuric acid solution (or other solution desired). Move the panel with the drops gently onto the pre-heated gradient surface for the 30 minutes.
- At the end of the 30 minutes, remove the panel and obtain the print-out of the actual temperature of each measuring point. Rinse off any remaining etching material with tap water. Using a facial tissue, wipe off any excess water. Spray the panel with "Super-Clear® lens cleaner" from AOSafety Products and wipe it off using a clean tissue.

  grade 1 - barely visible
  grade 2 - complete circle visible, but not filled in
  grade 3 - totally visible and filled circle
  grade 4 - blistering evident
  grade 5 - removal of film to bare panel

- Examine the panel under a good light source. Tilt the panel back and forth and note the temperature corresponding to the first visually etched spot, no matter how faint. Record the temperature for that point from the print-out. This value is the minimum spot temperature (MST). To make the faintest rings visible, exhale, as if cleaning glasses, over the rings where "grade 1" damage starts. (This spot may disappear from visual sight in a short period of time.) Grade each using a scale of 1 to 5, with 5 being the worst:
- Sum all the grades. This value is the total acid effect. A maximum of 225 is possible, if all spots etch through to the bare metal. To express acid resistance on a scale from 0.0 to 1.0, take this total number and divide it by the maximum (i.e., 97/225), and subtract the result from 1.0. This gives a scaled acid value [1.0-(97/225)]=0.57. A zero rating would be equal to a panel receiving a total acid effect of 225 (total failure) and a 1.0 rating would be equal to a panel not being affected by the acid at all.

It is possible to do multiple tests of several solutions at the same time.

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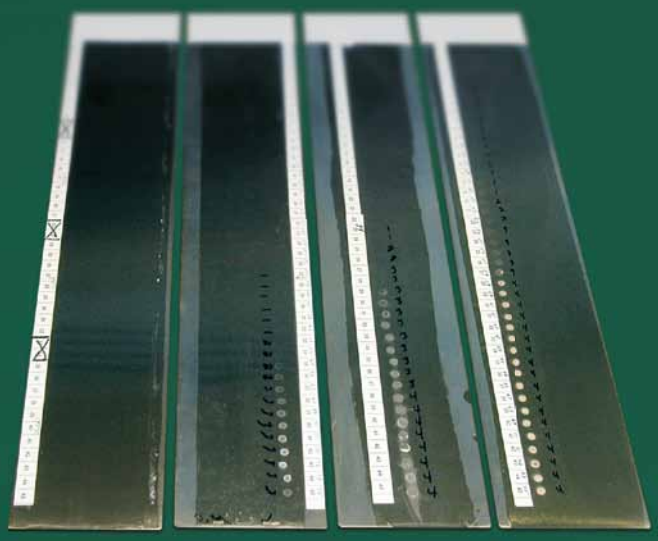

Figure 1

Figure 1 shows an ordered series of test panels. The most affected panels are on the right. In this study, the acrylic polyol used was Macrynal SM 515/70BAC. Three variables were examined. The DFT (dry film thickness), the ratio of melamine resin to acrylic resin, and the crosslinker type were examined. The coating formulations and the acid etch results are shown in Table 1.

Table 1 / Formulations and Results

Materials (g)				
Macrynal SM 515/70BAC	14.01	14.01	14.01	8.25
Resimene 747	3.27	2.45	2.45	-
Desmodur N 3300	-	-	-	3.01
2-heptanone (MAK)	1.66	2.26	2.26	-
Propylene glycol methyl ether	0.83	1.13	1.13	-
Xylene	-	-	-	3.14
pTSA (5.0% IPA/t-BuOH)	0.368	0.368	0.368	-
15 minute flash; bake 30 minutes at 138 °C				
DFT (mil)	1	1	2	1
72+ hours post cure				
Acid Spot Test Results:				
MST (°C)	49	53	52	53
Scaled acid value	0.609	0.703	0.711	0.622
Gradient oven - 30 minutes, heater zones set between 35-75 °C				

The acid-etch values found for the 80/20 (acrylic/melamine) ratio coating indicate that this ratio produced a more acid resistant coating than the 75/25 ratio coating. Notice that the 2 mil coating did not significantly improve the acid-etch resistance as compared to the 1 mil coating. This demonstrates that as little as a 1 mil coating can have good acid-etch resistance. When the two different crosslinkers were compared, the melamine-crosslinked coating produced a slightly better acid-etch-resistant coating.

This is not a surprising result. Melamine systems can be made to produce coatings with similar acid-etch resistance as isocyanate systems. The choice of the vehicle binder resin seems to be the key in producing a coating with improved acid etch resistance when using a melamine crosslinker. Acrylic resins modified with alkoxysilane groups or with carbamate functionality are two technologies used to increase acid-etch resistance.

Reference:

By George D. Vaughn, James B. Downie and Patricia E. Ferrell, Paint & Coatings Industry, page 74 - 76, August 2001

Conclusion:

The gradient-oven is a valuable tool for testing acid-etch resistance. This simple test saves valuable laboratory time in preparing panels and helps to improve coating properties. The test results show a good correlation to actual Jacksonville test data.

A great screening tool.

gradient-oven

see pages 225 - 234.