

Structure/Property Relationships In Flexible Alkoxysilane Automotive Coatings

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INTRODUCTION

The scratch resistance behavior of automotive coatings has been studied extensively using many different analysis techniques.^{1,2} The ability of a coating to withstand appearance degradation caused by mechanical stress under a specific set of conditions is still not widely correlated with the technique utilized or the coating material attributes. The shape, size, and geometry of the indenter utilized in the scratching event, the temperature at which the scratching occurs, and the rate and load at which the scratch indenter mechanically abrades the coating surface are all variables one must consider.³ Jarde¹ studied the scratch resistance behavior of several coatings utilizing different indenter geometries to identify three main scratch mechanisms. Elastic-plastic deformation was observed under mild abrasive conditions (e.g., where the indenter geometry angles are small or when the experiments are carried out with a Berkovich indenter). Irregular fracture processes occur when the attack angle is larger (e.g., with a cube corner indenter with its face in the direction of the scratch). When a cube corner indenter is used with its edge in the direction of the scratch, a regular longitudinal fracture propagates in front of the indenter. Indenter geometry therefore played a major role in the elastic-plastic deformation morphology of the coating analyzed.

Scratch resistance of coatings depends upon several factors, such as the chemical composition, the molecular weight of the resin backbone, the crosslinker type and crosslink density, the glass transition temperature, additives, cure temperature, etc. Coatings respond to mechanical stress in several ways, depending on the applied load. In the dynamic process of scratching, several tribological processes can occur, namely viscoelastic creep (time dependent processes of visco-plastic deformation and viscoelastic relaxation),⁴ strain-hardening, microcracking (fracture), and surface fatigue. If fracture occurs, no self-healing within the coating is evident over time. If the applied stress is of short duration and does not exceed the yield stress of the coating, it will undergo elastic deformation.

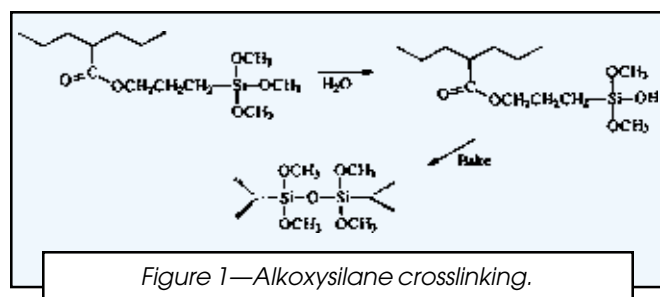
Flexible automotive coatings are susceptible to scratch and mar damage, especially during finishing and assembly operations. One-component (1K) flexible clearcoats exhibit very good scratch and mar resistance, but unfortunately suffer from poor durability and environmental etch resistance. Two-component clearcoats offer improvements in both etch and durability, but at the expense of scratch and mar. In this paper, the concept and properties of 1K flexibilized silane clearcoats for use on automotive plastics will be introduced and their structure/property relationships examined as they apply to scratch and mar.

The role of coating crosslink density, toughness, glass transition temperature (T_g), and surface profile on the scratch damage of coated plastic substrates will be described. In addition, a new scratch methodology, termed Scratcho, is utilized to determine relative scratch performance and is compared to conventional scratch resistance testing. Results to date indicate that hardness, as affected by the glass transition temperature, and crosslink density, as it contributes to higher essential work values, both affect resultant scratch propensity of the flexible coatings. The relative ranking of different coating systems employing alternate crosslinkers (e.g., isocyanate and melamine) is also presented and compared to the newly developed silane crosslinked coatings.

Presented at the 28th International Waterborne, High-Solids, and Powder Coatings Symposium, Feb. 21-23, 2001, in New Orleans, LA.

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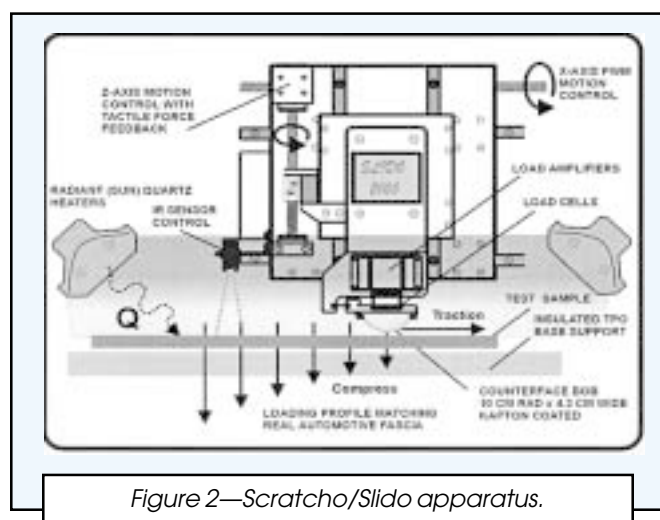
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tion and may be able to instantly recover any deformation upon removal of the stress. Although time-dependent properties of polymers have been studied, only a few have been reported for time dependence in mechanical characterization of surfaces.^{5,6} Viscoplastic properties are related to the polymer's sensitivity to deformation rate being applied to the surface. Load rate, achieved by varying the scratch tip velocity at equivalent penetration depths and indenter geometries, was found to affect scratch width.⁷ Faster motion resulted in a thinner residual scratch.

The overall scratch resistance of the coating was shown to be related to the time-dependence of its material properties. Jardret¹ found that relaxation phenomenon in prescratched clearcoats occurred through reduction of the "pile-up" height of the scratch while the scratch width remained constant.

Visibility of scratches produced within clearcoats remains a subject of much controversy. Ottaviani⁸ utilized goniometric techniques to determine the effect of incident angle of light on scratch visibility. Fractured surfaces were always visible, independent of incident light angle. Plastic deformations in the clearcoat samples, however, were only visible under certain lighting conditions. Jardet¹ found that if the scratch direction coincided with the observation and lighting direction, the scratch was not visible. When the sample was turned 90° however, the scratch appeared. Scratch morphology, therefore, plays an important role in visibility. With field scratches (those occurring in automotive parts under real world conditions) varying within the top 50 µm of the surface, our efforts focused on determining the effective load at which scratches were visible in automotive plastic coatings.



The increase of one-component rigid alkoxysilane clearcoats on automobile bodies has dramatically increased since their launch in the early 1990s due to their outstanding environmental etch, excellent scratch, and durability exposure. Alkoxysilane crosslinking chemistry is based on the hydrolysis of alkoxysilane to silanol and the condensation of two silanol groups to a silane bond with the evolution of water. The crosslinking, which results in very acid-resistant silicate bonds is in fact acid catalyzed as shown in Figure 1.

Rigid 1K alkoxysilanes offer many advantages over conventional melamine coatings including a durable hydrolytically resistant chemical bond, highly reactive multiple crosslinking sites that can react with themselves, low viscosity (low VOC), low toxicity, and one package technology.

We have explored flexibilizing this alkoxysilane crosslinked technology and have developed two generations of flexible alkoxysilane coatings for automotive plastics. Due to the high cost of the alkoxysilane functional materials, melamine was used as an auxiliary crosslinker to improve out-of-oven properties and reduce product cost in the flexible silane clearcoats. Additional melamine and auxiliary polyurethane-based crosslinker is added to the Generation[®] VI clearcoats. Table 1 describes the clearcoat coatings used in conjunction with black 1K basecoat over solventborne adhesion promoter on TPO.

Within this paper, we describe the mechanical attributes of one component alkoxysilane crosslinked coatings and compare these to the more traditional melamine crosslinked and isocyanate crosslinked coatings as related to surface damageability, namely scratch and compressive shear loading damage. We evaluate the effects of weathering on retained surface properties as well, simulating six-month exposure in the field. We have utilized conventional scratch techniques, namely the Ford five-finger scratch testing protocol, the ASTM AATC crockmeter test, as well as newly developed scratch techniques (a nanoscratch test and a macroscratch test protocol) to determine the resistance of the clearcoats. We relate these properties to selected mechanical attributes of the clearcoats, e.g., essential work of fracture, glass transition temperature, and microhardness.

EXPERIMENTAL

Coating Preparation

All coatings utilized in this work were spray applied (atomized air) to black 10 cm × 30 cm thermoplastic olefin (TPO) panels, baked 30 min at 127°C (part temperature), and post-aged 72 hr prior to testing. For samples utilized in any of the testing protocol, with the exception of those utilized in differential scanning calorimetry (DSC) analysis and essential work calculations, the panels were prepared by applying a solventborne adhesion promoter, a one-component solventborne basecoat, and the selected solventborne clearcoat (wet-on-wet-on-wet application), with dry film thicknesses of 8 µm, 20 µm–37 µm, and 37 µm, respectively. The clearcoats tested were formulated from one of the following solventborne chemistries, and

Table 1—Selected Properties of Clearcoat Compositions

Coatings	Hardness (N/mm ²)	Swelling	T _g (°C)	W _{ess} (nJ)	Nanoscratch Fracture Response (mN)	Crockmeter % 20° Gloss Retention	Ford Five-Finger Scratch Material Displaced (microns) initial	Scratcho (kg) before Weathering (WOM)	Slide Traction Force (kg) before WOM
CC1	23.6	1.438	43.4	2.33	19.83	67	0.83	3.4	112.5
CC2	60.1	1.554	50.9	16.33	18.15	85.5	0.63	4.5	86.5
CC3	58.3	1.431	49.5	9.22	11.95	81	0.55	4	122
CC4	33	1.472	42.4	5.45	20.7	80.5	0.59	3.9	95.6
CC5	70.1	1.42	53.4	4.43	8.47	73	0.71	3.5	114
CC6	23.8	1.446	43.7	5.02	11.52	77	0.93	3.6	115
CC7	37.8	1.382	47.2	2.13	16.25	72	0.81	3.3	98.7
CC8	24.3	1.437	45	0.33	23.83	53.5	1.19	2.8	80.2

all subsequent nomenclature utilized in this paper will be directed to the clearcoat (CC) number, e.g., CC1:

- CC1 1K Flexible Melamine Control
- CC2 2K Flexible Isocyanate Control
- CC3 1K Flexible Silane Clear (20% flexibilizer)
- CC4 1K Flexible Silane Clear (35% flexibilizer)
- CC5 1K Generation® IV Rigid Clear Control
- CC6 1K Generation® VI Flexible Clear (12% flexibilizer)
- CC7 1K Generation® VI Flexible Clear (22% flexibilizer)
- CC8 1K Generation® VI Flexible Clear (32% flexibilizer)

Coatings were applied at conventional film builds (in microns) (5-7.5 AP, 15-20 BC, 45-50 CC), baked for 25 min at 260°F (part temperature) and post-cured at ambient for a minimum of 72 hr prior to testing. Clearcoat only panels were sprayed on TPO and then peeled off for mechanical property testing.

Nanoscratch Single Indenter Technique

Nanoscratch measurements provide a means of obtaining quantitative information on coating mechanical response to surface forces, and on transitions in response from elastic recovery, to viscoplastic deformation, and eventually to fracture. Normal force at the fracture threshold and plastic flow resistance are key coating parameters that correlate with scratch and mar, as well as other coating attributes. This method is a very sensitive measure of the mechanical property changes that take place during

exposure and clearly shows the difference in character of the two types of crosslinking chemistry used.

Single Indenter Nanoscratch

To understand the mechanism of mar damage, a nanoscratch technique was developed to quantitatively study scratch and mar behavior of coatings. In a typical nanoscratch experiment, a well-defined indenter (i.e., in shape, size, position, and material) was controlled to penetrate into a coating surface in a vertical position while the coated panel was moved in a horizontal direction.⁹ During this process, indenter displacement (in nanometers), as well as normal and tangential forces (in micro-Newtons), were continuously monitored. The damage events were recorded by use of an optical microscope equipped with a video camera having video capture capabilities. The controllable parameters of the nanoscratch experiment are penetration rate, scratch rate, lubrication condition, cutting geometry and coating temperature. Our experiments employed a ramping function technique where indenter penetration increased continuously while the panel moved horizontally. This technique is very useful for studying the wide range of fracture and plastic flow conditions that were found in these experiments.

A complete experiment consisted of the following: (1) a prescratch to measure the topography of the undamaged coating, (2) a scratch to produce the damage, and (3) a postscratch to determine the damage. The indenter followed the same path for all three scratches. A constant normal force of 20 μ N was used for the pre- and postscratch. The normal force was increased linearly at a rate of 0.1 mN/sec to the limit of the experiment. The indenter used was a diamond cone with a 60° angle and a tip radius of 3 μ m. Scratch rate was constant at 25 μ m/sec. All scratch experiments were done at 21°C.



Figure 3—Helix scratch head utilized in Scratcho.

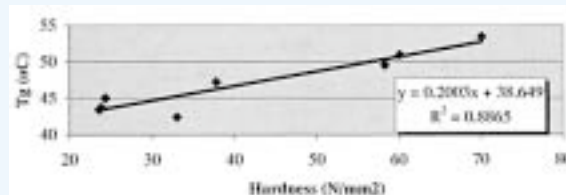


Figure 4—T_g vs. hardness.

Table 2—Damage Resistance Behavior of Clearcoat Compositions

Coating	Slido Traction Force (kg) before WOM	Slido Compressive Force (kg) before WOM	Slido Traction Force (kg) after WOM ^a	Slido Compressive Force (kg) after WOM ^a	Scratcho (kg) before WOM	Scratcho (kg) after WOM ^a
CC1	112.5	636.3	139.5	45.5	3.4	3.2
CC2	86.5	223.4	154.9	45.5	4.5	4
CC3	122	459.1	154.9	45.5	4	2.5
CC4	95.6	188.9	168.2	45.5	3.9	3.4
CC5	114	592.2	118	45.5	3.5	2.1
CC6	115	641.3	125.6	45.5	3.6	2.1
CC7	98.7	391.7	135	45.5	3.3	3
CC8	80.2	223.4	78.1	45.5	2.4	2.3

(a) After 250 kJ Xenon Arc Weatherometer (WOM) exposure.

Macro-Multiple Indenter Scratch

“Scratcho” results were obtained on a commercially available Slido apparatus (shown in Figure 2) equipped with a scratch head comprised of a stainless steel helix, where each helix head of the 15 heads comprising the helix is approximately 0.8 mm in diameter, with 2 mm between each helix head (Figure 3). In this scratch methodology, the painted sample is placed onto an insulated TPO base support and the sample is heated to 68.3°C by means of radiant quartz heaters, maintained at temperature with an infrared sensor control. The helix scratch head is then loaded onto the sample at a ramped load rate of 4.5 to 136.4 kg over a distance of 15.24 cm, with an acceleration of 50.8 cm/sec² and a velocity of 5.08 cm/sec.

Scratch deformation imparted to the painted panel is analyzed under a McBeth white light at a 45° angle. The first sign of fracture within the paint is reported as a load function, e.g., kilograms (kg) to first fracture. Painted panels were also exposed to modified SAE J1960 conditions in a Xenon Arc weatherometer equipped with borosilicate/borosilicate inner and outer filters. Exposure time was 250 kJ. The scratch testing methodology was reproduced on

the weathered (WOM) panels and the pounds to first fracture reported.

Compressive-Shear Damageability

“Slido” testing was performed on the apparatus described in Figure 2. As shown in the figure, a painted panel is placed under the “bob” (an aluminum counterface with a 10.2 cm diameter) covered with a polyimide film, and the bob is translated across the surface of the panel at a preset loading rate with a known velocity and acceleration. The temperature of the plaque can be maintained at above ambient temperatures through use of infrared heaters located in the apparatus. Resultant plots are obtained of load versus displacement, through which the coefficient of friction of the coating can be calculated. Values obtained from the test plot also include the compression force (kg) to failure, defined as the force exerted perpendicular to the coating, and the traction force (kg) at failure, defined as the force exerted parallel to the coating. Actual conditions utilized in the Slido testing were as follows: 15.3 cm run length (defined as the distance traversed by the bob on the panel); acceleration of 50.8 cm/sec², velocity of 5.1 cm/sec, and a temperature of 68.3°C.

The essential work values were obtained on each clearcoat-only system by methods described previously¹⁰ on an Instron 5565 electromechanical testing apparatus. Tensile tests were performed at 25°C and 50% relative humidity. The displacement rate was 0.033 mm/sec. Clearcoat-only films were prepared as discussed above, peeled from the TPO substrate, and cut into gage lengths of 25.4 mm. Double-edged notch tension specimen geometry of different ligament lengths was utilized. The notches were made perpendicular to and at the mid-gage length using a razor blade with a tip radius of 0.01 mm. To obtain sharp crack tips, the razor blade was drawn from the inside of the notch to the outside edge of the gage length. Three specimens each of five different ligament lengths were tested for each clearcoat. The ligament lengths were chosen to maintain plane strain conditions within the specimens. Essential work values were calculated by plotting the ligament length (x-axis) versus the work of fracture (area obtained under the stress-strain plot) (y-axis) and extrapolating the best straight line through the data points back to the zero-ligament length. The correlation coefficient to straight line goodness of fit, *r*², is also reported.

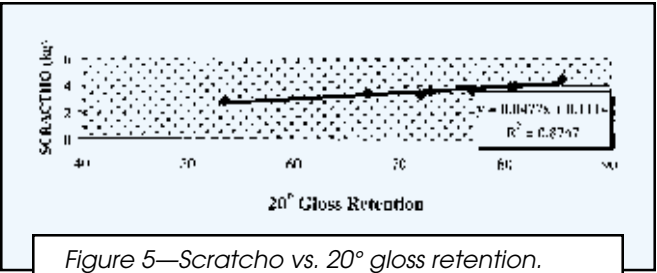


Figure 5—Scratcho vs. 20° gloss retention.

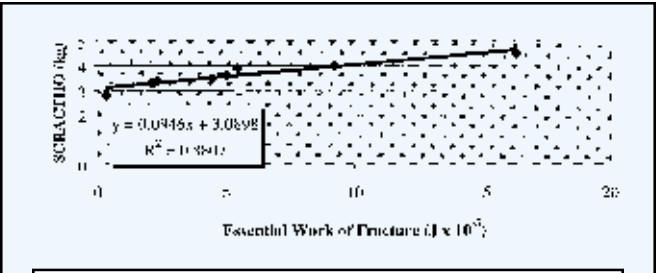


Figure 6—Scratcho vs. essential work of fracture.

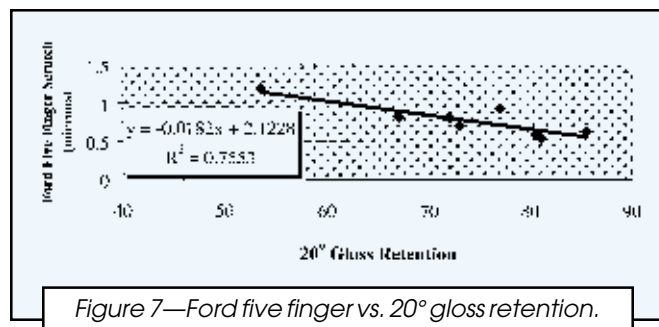


Figure 7—Ford five finger vs. 20° gloss retention.

Ford five-finger (FLTM 108-13) scratch ratings were obtained at room temperature (25°C) on adhesion promoted/basecoat/clearcoat TPO composites with the 7N finger. In this method the coated panel is placed onto a moveable platen onto which is placed a beam containing the scratch pin. The beam is 250 mm long and is equipped with a scratch pin that consists of a highly polished steel ball (1 mm ± 0.1 mm in diameter). The beam is driven by compressed air to draw the pin across the surface of the coated plaque to generate a scratch. Sliding velocity was maintained at approximately 100 mm/sec. Measurements of the scratch "ditch" depth and scratch "shoulder" threshold, together termed material displaced, were obtained with a Wyko interferometer at a magnification of 5×.

Microhardness measurements on the basecoat/clearcoat composite structures were made with a Fischer Microhardness H-100 apparatus equipped with a Vickers indenter and a 100 mN load. The load rate was applied in 60 steps, with one second between steps. Unloading was accomplished after seven seconds of creep in 60 steps, with one second between steps. Values reported include the plastic hardness, H_{plas} , which is a measure of the plastic deformation component of the indentation.

Gloss retention of marred surfaces was measured with a 20° gloss meter and reported as a percentage of the initial unmarred surface gloss. Marring was performed on an AATCC crockmeter equipped with a cloth pad (DP-cloth, HQ manufactured by Struers, Copenhagen, Denmark and distributed by VWR Scientific) fitted with 0.01 gram of 63 micron (220 grit) alumina oxide. The load was kept constant at 940 grams, the sliding velocity maintained at approximately 21 mm/sec, and a total of two double rubs were performed on each sample.

To determine the crosslink properties of each clearcoat, the coating was placed in methylene chloride and the swelling characteristics were calculated. In this procedure, a free film of the clearcoat is placed between two layers of aluminum foil. Using a Ladd punch, the film was cut to a disc of about 11 mm in diameter. The free film was then separated from the foil and placed into a microscope slide that was subsequently covered with a cover glass. Using a Leica microscope equipped with a 10× to 25× magnification lens and a graduated reticule, the initial diameter of the clearcoat was measured. Three drops of methylene chloride was placed between the two pieces of glass to swell the clearcoat. After five seconds (or when the clearcoat had reached a constant swelled diameter) the sample diameter was remeasured. The following calculations were made and the Area Swell is reported in Table 1.

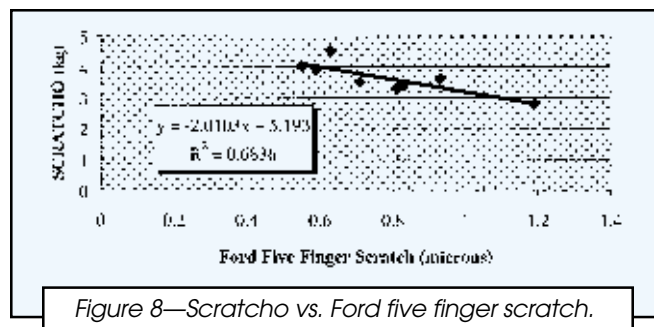


Figure 8—Scratcho vs. Ford five finger scratch.

1. Area Before: (length before solvent)²
2. Area After: (length after solvent)²
3. Linear Swell: (linear length difference/length before solvent)*100
4. Area Swell: 100* (area difference)/area before solvent
5. Area Difference: area after solvent–area before solvent

Glass transition temperatures of each clearcoat were determined on a DuPont 2200 differential scanning calorimeter equipped with version 4.0B software. Sample size of two to seven milligrams was heated at a rate of 5°C per minute, with a scan range of –75°C to 250°C.

RESULTS AND DISCUSSION

All data obtained in the series of testing described *vide supra* are listed in Tables 1 and 2. For the sake of data manipulation and ease of discussion, the data tabulated in Tables 1 and 2 will be discussed in terms of best fit to linear relationships achieved through charts depicted in Figures 4 through 10.

As can be viewed in Figure 4, the T_g has a direct effect on the microhardness of each clearcoat studied, with the goodness of fit reaching 88% to linear behavior. Those coatings with a higher T_g exhibit a harder surface. It is important to note that the microhardness values indicated in Table 1 and depicted in Figure 4 are representative of the top 10–15 μm of the coating. Since all of the scratch testing described later in this section resulted in damage patterns in the 1 to 2 μm depth range, the microhardness of the coating "surface" is representative of the coating's ability to withstand applied stresses. The hardest coatings after the 72 hr post-cure prior to test were Generation VI rigid, 2K Isocyanate, and the Gen IV (with 20% flex resin added), while the softest coatings were the flexible melamine control and the Gen VI clearcoats (containing 12% and 32% flexibilizing resin).

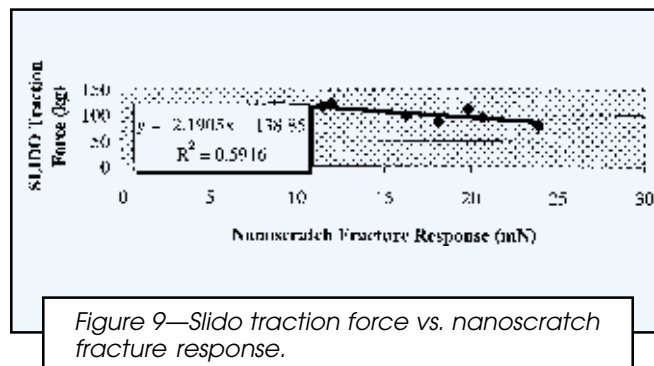
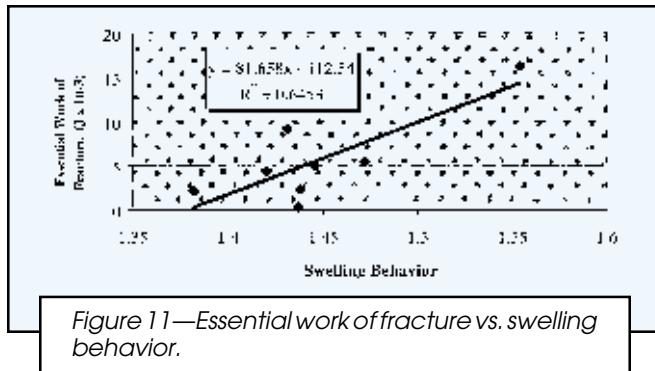
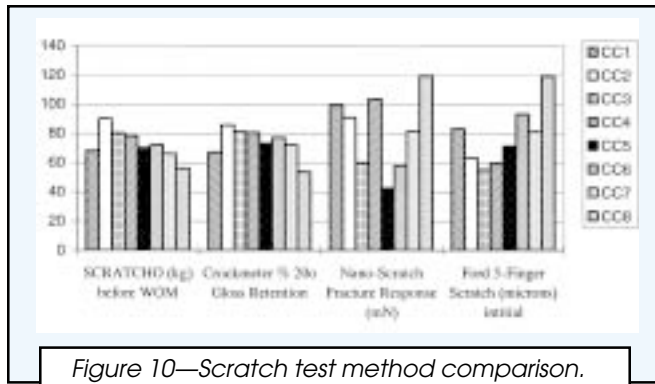
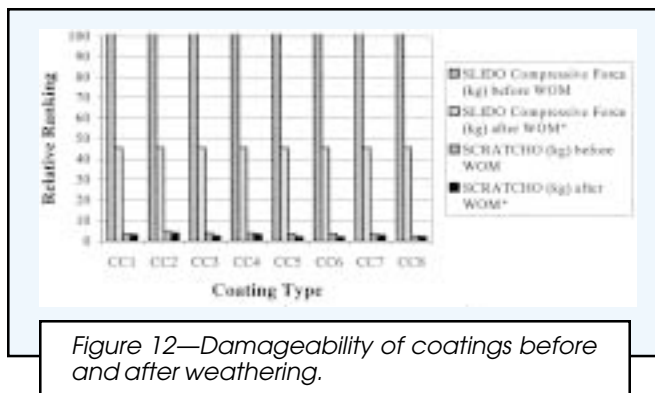


Figure 9—Slido traction force vs. nanoscratch fracture response.



The force at which visible macro-fracture was first observed in Scratcho testing is compared to the gloss retention achieved in the crockmeter testing, showing a reasonable linear correlation (r^2 of 88%) (Figure 5). The coatings exhibiting the best macro-fracture resistance, as determined from the Scratcho macro-multiple indenter procedure (and correlated with the AATC crockmeter results) were the 2K Isocyanate and Generation IV Silane Clear (35% flexibilizing resin added) clearcoats while the worst clearcoat for fracture resistance appears to be the Generation VI rigid (with 32% flexibilizing resin added) clearcoat. It should be noted that the 2K isocyanate clear used was a formulation with dramatically improved scratch resistance, which is not typical of most commercially available 2K products.

When attempting to explain the cause of better fracture resistance of one clearcoat over another, we can compare the essential work of fracture to its resultant scratch resis-

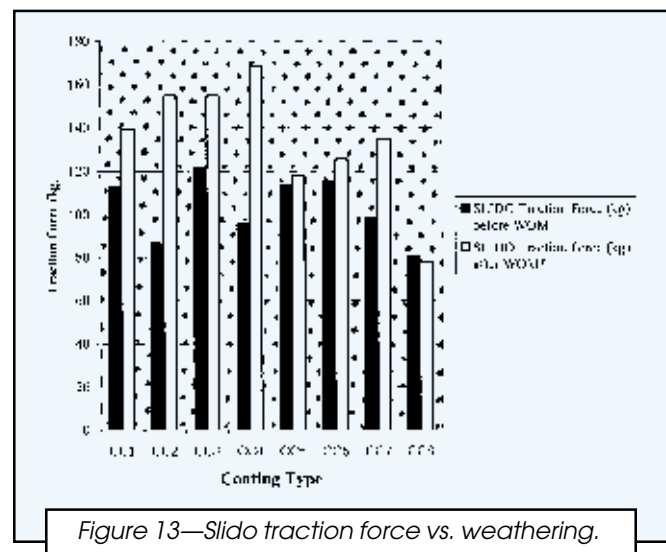


tance (Figure 6). There appears to be a very good fit to linear behavior (r^2 of 88%) between the essential work of fracture and the resultant macro-fracture resistance of the coatings studied, suggesting a possible cause. It does correlate that the “tougher” a coating system becomes (as evidenced by higher essential work of fracture values), the better chance it has to withstand an applied load without fracturing.

Ford five-finger scratch resistance is compared to AATC gloss retention (Figure 7). Both methods can be considered “micro-scratch” techniques due to the low loadings utilized in the testing protocols. As shown in Figure 7, the two results correlate fairly well to one another, with a 75% goodness of fit to linear behavior. As depicted before in Figure 6, when correlating the ability of the coatings to withstand fracture, the 2K Iso and the Gen IV (with 35% flexibilizing resin) coatings perform the best for fracture resistance (with the Gen IV with 20% flexibilizing resin also performing well), while the Gen VI rigid coating with 32% flexibilizing resin performs the worst. The weaker correlation in Figure 6 relative to Figure 5 may be due to differences in loading in the two test protocols. While the Scratcho testing protocol uses the higher loadings, it may be more representative of higher load scratch deformations resulting from deformations “deeper” in the coating composite versus those of surface deformation within the top few microns of the coating.

When attempting to find correlation between the “macro-scratch” response as evidenced in the Scratcho testing and the “micro-scratch” responses evidenced in the Ford five-finger and the nano-single indenter testing methodologies, the goodness of fit to linearity is sacrificed (Figures 8 and 9). Although the general relationships derived above for “best” and “worst” coatings for resistance to fracture generally hold, the variation in response may be due to nonhomogeneous material attributes throughout the depth of the coatings. This scenario is purely a speculation based on prior literature¹¹ since this study did not attempt to depth profile material attributes of the coatings evaluated.

Figure 10 compares the results obtained from these different scratch test methods and has the data adjusted to



the same magnitude so that all four methods can be compared on one chart. As can be seen from *Figure 10*, the correlation between Scratcho and the crockmeter is relatively close while the correlation between the nano-indenter and the Ford five-finger scratch shows no consistent trend.

The last correlation made in attempts to relate material attributes to one another is shown in *Figure 11*. It appears from the data, although the fit to linearity is not optimal (65% goodness of fit), that as the swelling behavior of a coating decreases the essential work of fracture decreases. Intuitively, this makes sense since a more highly crosslinked network will be more subject to fracture and a resultant decrease in essential work than a coating with less crosslink density, exhibiting more swelling behavior. The error or lack of optimal fit to linearity between the two parameters may be due to the variances in molecular weight between crosslinks that can also contribute to the swelling behavior of a coating system.

It is interesting to note that the damageability of the coatings to scratch (Scratcho) and compressive shear loading behavior (Slido) before and after weathering differ (*Figure 12*). The resistance to scratching and compressive shear loading events decrease after 250 kJ of weatherometer exposure, regardless of coating type. This is presumably due to an increase in crosslink density with a resultant increase in coating brittleness.

As viewed in *Figure 13*, however, the traction force of the coatings after exposure increases. According to Ryntz, et al.¹² this is not surprising since the hardness of the coating can influence the ability of a coating to distribute applied stress. As the coating becomes harder, presumably due to the increase in crosslink density of the coatings upon further aging in the weatherometer, the dissipation of applied stress increases resulting in an increase in traction or lateral force across the substrate. From this data, it appears that the 2K isocyanate and the Gen IV (with 35% added flexibilizing resin) coatings are best at withstanding applied stress after weathering, while the Gen VI rigid coating (with 32% added flexibilizing agent) is the worst.

SUMMARY

Scratching of automotive clearcoats continues to be an issue with today's automobiles. While test protocols differ in their applied loading, depth penetration, and ultimate fracture mechanism, work continues to explore these various methods to better understand their correlation with real world scratch damage. The development and proper

formulation of one component flexible alkoxysilane coatings can offer excellent performance with some of these test protocols. The flexible alkoxysilane coating compositions formulated for plastic possess high essential work of fracture values which in turn result in very good micro- and macro-scratch performance, e.g., Ford five-finger/nano-scratch results and Scratcho/crockmeter results, respectively.

The results obtained with the newly developed Scratcho test methodology correlate very well with the more traditional crockmeter results whereas the damage produced with the Ford five-finger scratch is fairly similar to that produced in the nano-scratch fracture response. It is important to note that applied stresses in the field can vary, therefore the ability of the coating to withstand both micro- and macro-scratch events is imperative. We believe that with the newly developed testing protocol described previously we are able to aptly predict scratch resistance of selected coatings. In turn, the correlation of the physical/mechanical properties of the selected coating to the resultant scratch propensity allows one to develop more scratch resistant coatings in the laboratory which will result in lower scratch damage in the field.

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