Tech Bulletin

Cure Testing of DT-420 at Ambient Temperature vs. High Temperature

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12/27/2017

Cure Testing of DT-420 at Ambient Temperature vs. High Temperature on Aluminum Panels

Cure testing on aluminum panels (native aluminum oxide surface) was performed at 20 °C by adding 500 microliters of DT-420 to the aluminum panels. The DT-400 series of coating compositions (including the DT 420 product featured here) are covered under U.S. 9,856,400 issued January 2, 2018. Coatings formed with the DT-400 series are the subject of pending U.S. Patent Application No. 15/820,853 filed November 22, 2017.

Before application, the aluminum panels were cleaned with acetone and incubated at 120 °C for 24 h to remove solvent residue. The samples for studying ambient temperature coating were incubated at 20 °C (20% rel. humidity) for 5 days. Fourier transform infrared (FTIR) spectroscopy of the coatings on alumina was measured within 5 minutes of coating and then again at 6 h, 12 h, 24 h, 48 h, 72 h, 96 h and 120 h. A second set of samples were coated at 20 °C as described above and then incubated at 120 °C. FTIR spectroscopy was measured at 30 min, 1 h, 2 h, and 4 h.

Identification of a Signature FT-IR Peak to Quantify Curing Progress

DT-420 contains a Si-H functional group which reveals the chemical processes during curing. Figure 1 below shows the FTIR spectrum of DT-420 at 5 minutes after coating on aluminum at 20 C°. The Si-H absorption band is visible and labelled around 2100 cm^{-1}. The degree of coating's cure corresponds to the disappearance of the Si-H band. Once all the Si-H groups have reacted, the chemical curing stopped. The Si-H group appears around 2100 cm^{-1} in the FTIR spectra.

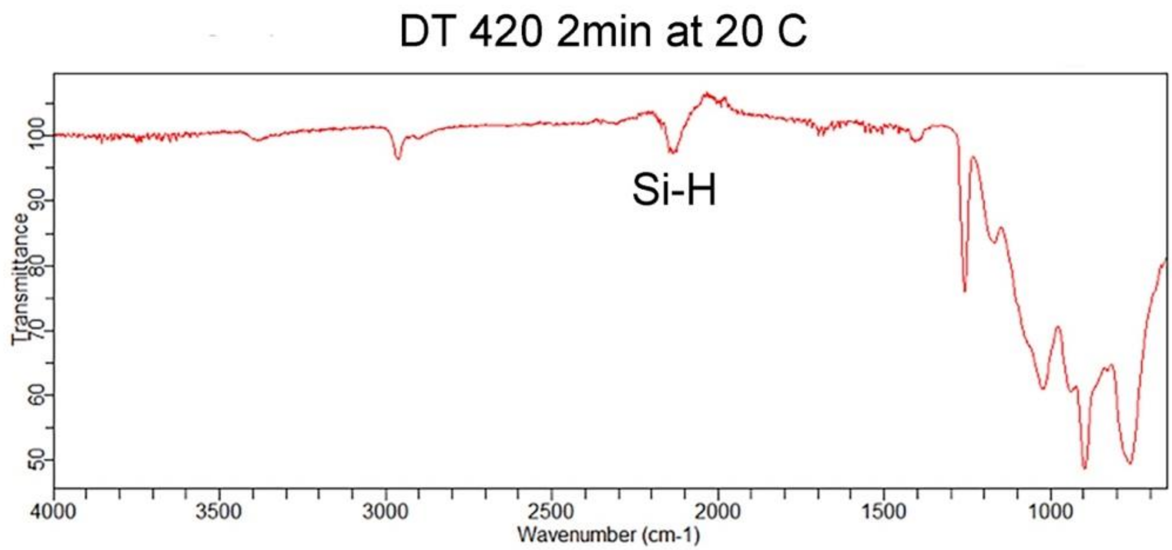

Figure 1: FTIR spectrum of DT-420 coated aluminum surface at 5 minutes after coating. Estimated film thickness is 10 microns.

Other FTIR peaks are the C-O stretch at 1250 cm^{-1}, C-N stretch around 1150 cm^{-1}, Si-O stretch around 1100 cm^{-1}, Si-N stretch around 900 cm^{-1}, and NH_4Cl absorption peaks (from Durazane™) around 775 cm^{-1}. Although it is difficult to follow the reaction progress from the FTIR peaks between 1250 and 900 cm^{-1}, these bands permit the comparison of polysiloxazane networks formed during thermal versus ambient curing. The quality of both coatings is compared based on their FTIR spectra.

The same behavior is found for DT-420. The FTIR peaks at 2200 cm^{-1} and 2000 cm^{-1} are typical for the Al-O stretching frequencies of aluminum oxide (Al_2O_3) in alumina. They are not from Si-H groups, which appear in the middle of the spectrum around 2100 cm^{-1}.

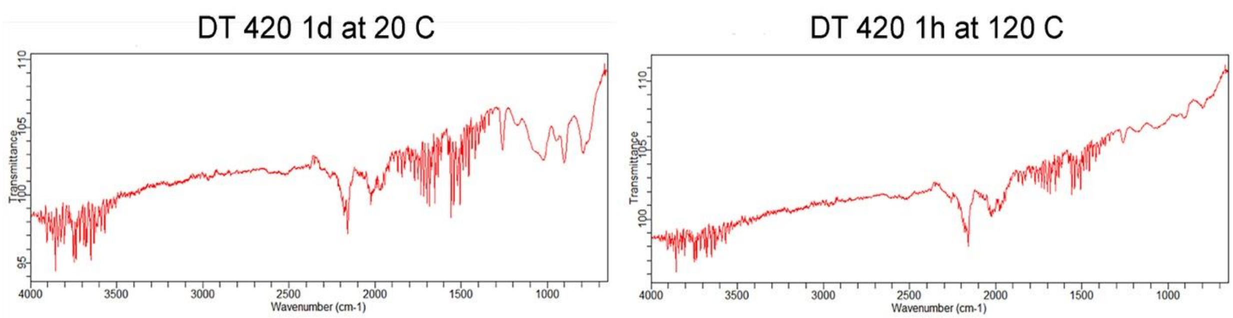

Figure 2: FTIR spectra of DT-420 on aluminum. Left: 24 h at 20 °C / 70 °F, film thickness of 5 microns. Right: thermal curing, 1 h at 120 °C / 250 °F, film thickness of 4 microns.

Chemical Reactions During Ambient and Thermal Coating Reactions of DT-420

Two different reactions are discernible: 1) Oxygen-mediated radical polymerization and crosslinking reactions and 2) thermal reactions. The DT-420 formulation triggers oxygen-mediated reactions, which contributes about 70-80% of its curing reactions, while thermal reactions constitute the remaining 20-30%. However, thermal reactions form the covalent bonds between the coating substrate and the coating. Common to all DT-420 crosslinking reactions are their consumption of Si-H bonds. For this reason, the curing progress can be followed by monitoring Si-H bands in FTIR.

Kinetics of the Ambient and Thermal Coating Reactions of DT-420 on Aluminum Test Surfaces

As mentioned above, the measured kinetics of Si-H band disappearance indicates the progress of the many curing reactions. For baseline, the original size of the Si-H band (measured 5 min after coating) indicated 0% curing. Conversely, 100% cured is defined when the Si-H band is completely gone.

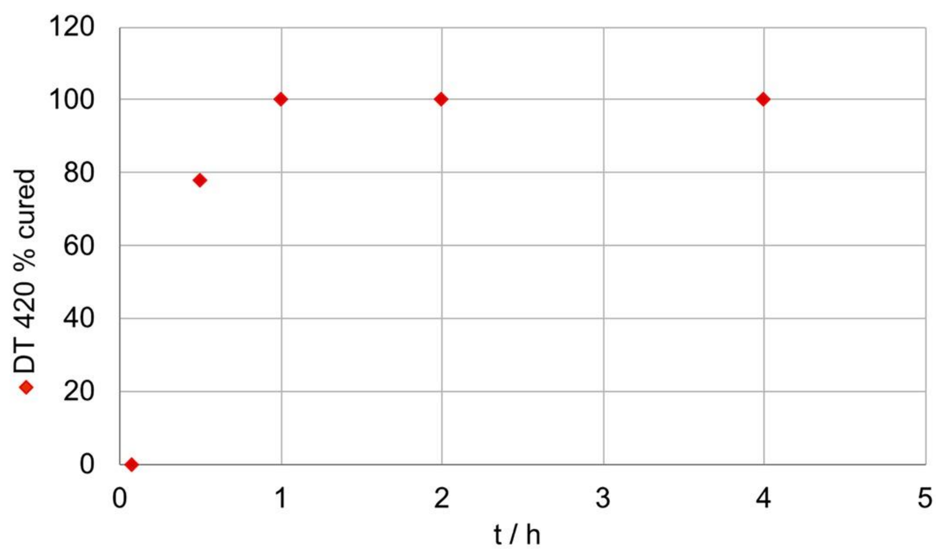

Figure 3: Thermal Curing on Aluminum. DT-420 is completely cured after 1 h at 120 °C/250 °F regardless of the film thickness.

Because DT-420 is dry to the touch within 15 min. of curing under these conditions, thermal curing can be safely stopped after 30 min. As FTIR analysis indicates, principally the same coating is formed during thermal and ambient curing, albeit per different kinetics.

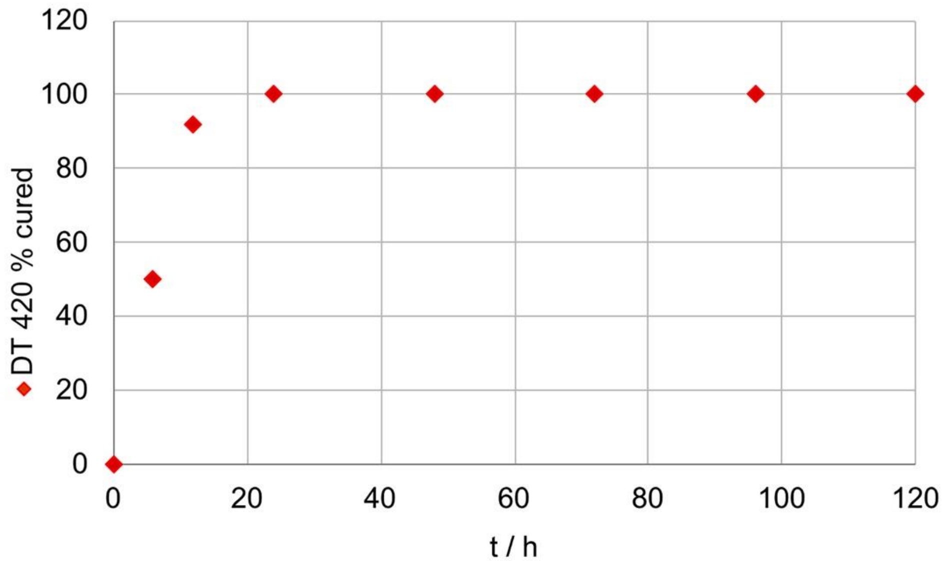

Figure 4: Ambient Curing on Aluminum. DT-420 reaches full cure after 1 h at 20 °C / 70 °F, ignoring film thickness. All formulations were dry to the touch after 55 ± 5 min of curing under these conditions.

Kinetics of the Ambient and Thermal Coating Reactions of DT-420 on Q-Panel Steel Test Surfaces

A: THERMAL CURING

DT-420 is completely cured after 1 h at 120 °C / 250 °F with film thickness of 5 microns. Because it was dry to the touch after 15 min of curing under these conditions, it is safe to stop the thermal curing after 30 min per FTIR analysis. When aluminum is the substrate, the same coating is formed during thermal and ambient curing, albeit per different kinetics.

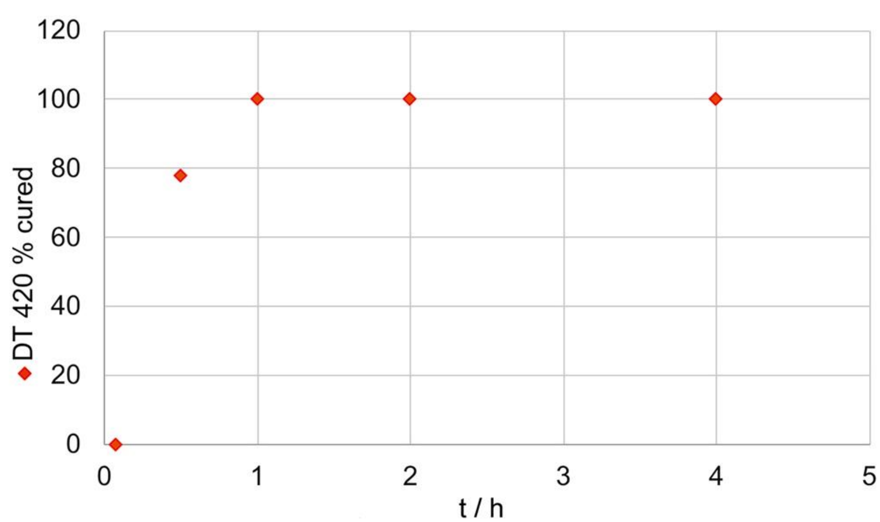

Figure 5: Thermal Curing of DT-420 on Q-panels

B: AMBIENT CURING

DT 420 was cured after 1 h at 20 °C / 70 °F at a film thickness of 5 microns. The formulation was dry to the touch after 50 ± 5 min of curing under these conditions. The coating is safe for storage etc. after 24 h of ambient coating.

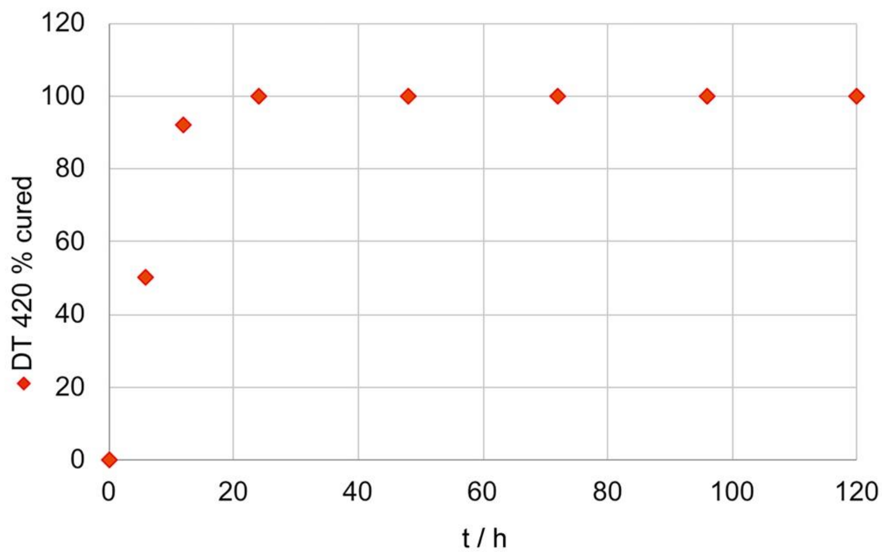

Figure 6: Ambient Curing of DT-420 on Q-panels

Re-Coating DT- 420

The DT-420 formulation does not permit for a second coat to be applied once the first coat has been fully cured. Exceptions to this are:

- If fully cured DT-420 has been abraded, scratched and/or wasn't applied <u>properly</u> the first time; leaving the surface with porosity. In these cases, the second application of DT-420 over the cured film will seek out and adhere to wherever those mechanical voids exist.

- The other exception is during the first 5 ± 2 minutes of ambient curing. During this period, the addition of more coating material is possible, leading to a homogeneous and slightly thicker coating.

Beyond the exceptions noted above, once the cross-polymerization of DT-420 has begun (> 5 min), a hydrophobic surface is formed, as indicated by the water droplet contact angles on the formed coatings of 105 ± 5 degrees.

The compositions themselves are more hydrophilic than the coatings they form, due to the changes the surface which prevents the second (DT-420) coating layer from spreading out. Droplets on the hydrophobic surface harden when exposed to the oxygen-containing atmosphere. No significant differences in recoating behavior were found on aluminum, cold-rolled steel, quartz, and nylon. Therefore, we conclude that the observed re-coating behavior is universal among these samples. It depends on the coating formulations and the sequence of coating but not on the substrate.

Conclusions from FT- IR Analysis

1) The chemical structure of DT-420 are the same, whether cured by thermal or ambient means.
2) Oxygen is necessary, because about 70-80% of the Si-H groups react in radical reactions, which depend on oxygen.

These studies have established that DT-420 can be successfully coated under ambient or thermal conditions, or combinations thereof, which makes it very versatile. DT-420 coatings are safe for storing solids, aqueous liquids and many organic solvents after 24 h of ambient coating. For an analysis of DT-420 coating stability at sustained elevated temperatures (>200 °C), please refer to the Dyna-tek Technical Bulletin entitled "Thermal Analysis of DT 420," dated May 16, 2018.