

Surface Wet Chemical Treatment to Improve the Adhesion of Teflon

Summary

A wet chemical surface treatment process has been developed to modify Teflon surface to improve their adhesions to epoxy adhesives. The results show that after 30 seconds submerging in wet chemical solution, the Teflon surface exhibits hydrophilic nature. The surface molecules contain hydroxide and carbonyl functional groups. Its adhesion to EP1640, a low CTE epoxy adhesive from United Adhesives, is significantly improved. The peeling strength reaches to a level higher than that of a regular PVC tubing to epoxy, and the peeling interface shows 100% cohesive mode between the modified layer and the bulk Teflon.

Experimental

Objective: This experiment is to establish a wet chemical treatment technique to modify Teflon surface, and to achieve a robust adhesion of Teflon tubing and electrical wire sleeve to epoxy adhesive. Several other surface treatment techniques have also been applied, and their adhesion results are compared.

Teflon Specimens: Teflon tubing and wire sleeve

Treatment Methods: Wet chemical developed in United Adhesives, Inc. Alternative methods include organic base wet chemical, plasma, and UV Ozone.

Adhesion Measurement: Instron 90 degree peeling adhesion

Surface Characterization: FTIR Spectrometry, Microscope, and wetting of water droplet.

Adhesives: Two adhesives were applied in this experiment – EP1640 low CTE epoxy from United Adhesives, and a regular epoxy (high CTE) from Supplier X.

Wet Chemical Procedure

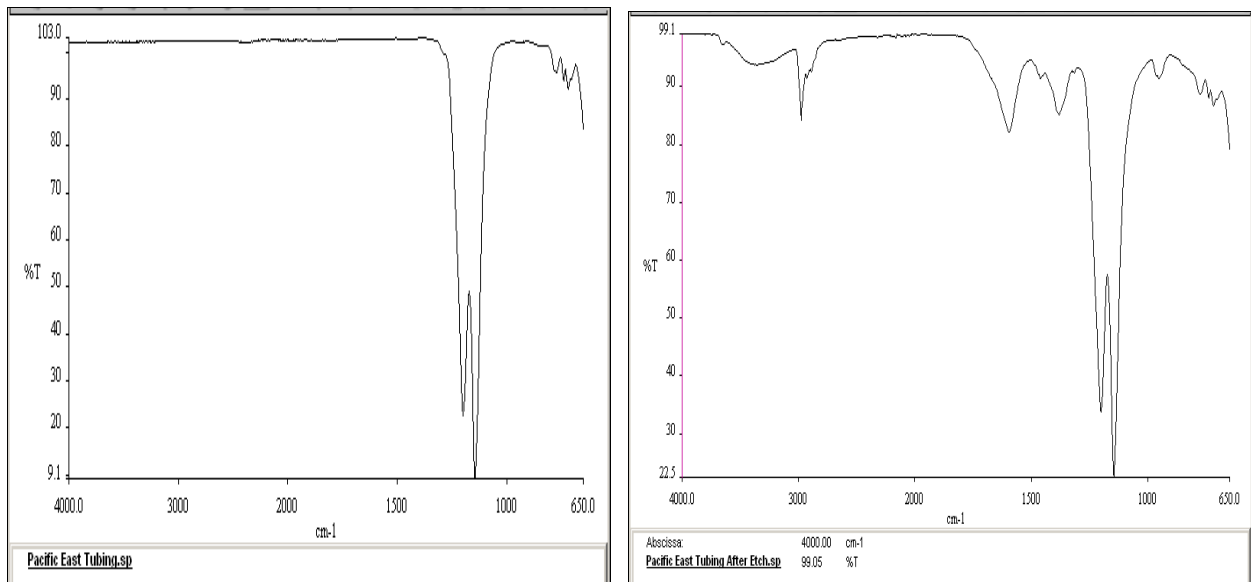
The Teflon tubing was cut into pieces with ¼ inch wide and 2 inch long. The Teflon wire was used as received. The submerged time was controlled either for 30 seconds or 60 seconds. After the submerging, the specimens were subject to Solution I and Solution II and then dried by blowing with air.

It was observed that, after the wet chemical process, the color of the tubing and the wire becomes dark. This color change is compared in following two photos. In each photo, the specimens on left are before the treatment, and on the right are after the treatment.



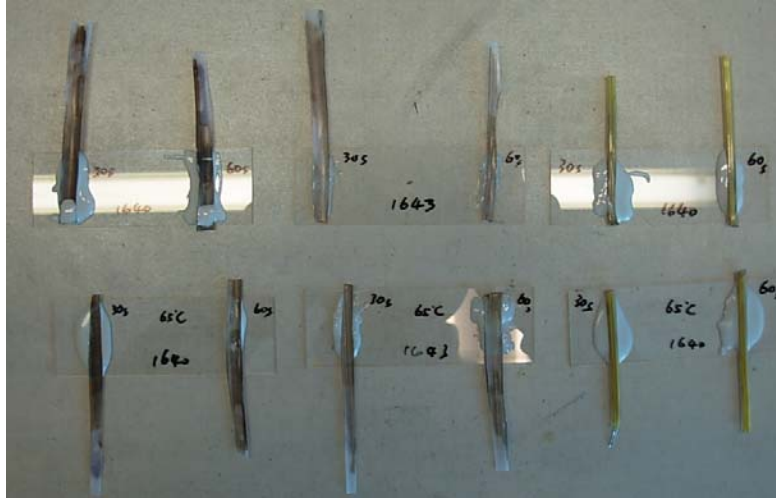
After the treatment, a water droplet test shows that the surface hydrophobic property has been changed to hydrophilic nature with a much better wetting of water.

A scanning of FTIR spectra of the Teflon tubing after the wet chemical treatment indicates that there are significant amounts of OH, C=O, and CH_x functional groups appearing on the surface. The two FTIR spectra are shown below. The left one is before the chemical treatment and the right one is after the chemical treatment.

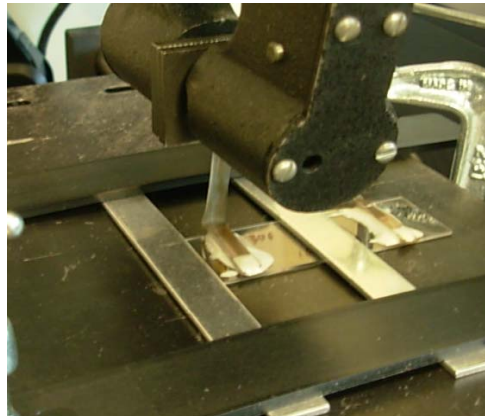
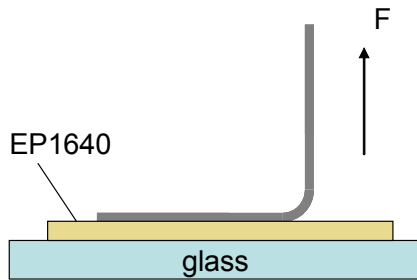


Quantitative adhesion measurements were performed with an Instron 90-degree peeling method. The procedure is:

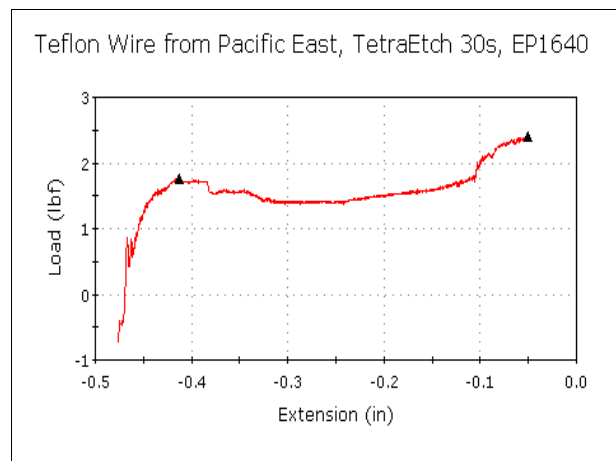
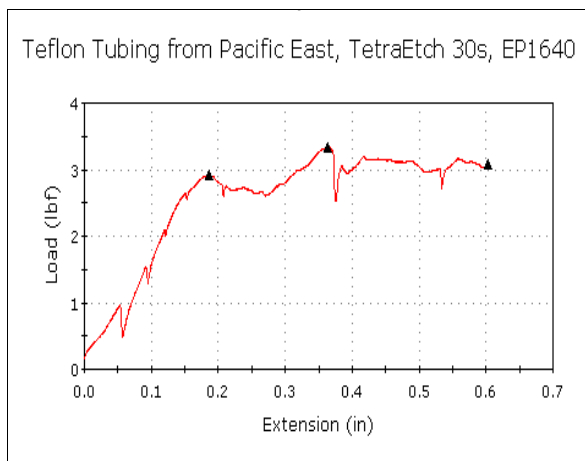
- a) Dispense epoxy adhesives on glass slides. Apply the tubing and wire specimens, treated or not treated, onto the adhesives, and cure either at ambient for 24 hrs or 65°C for 2 hrs. The specimens after the preparation are as shown in following photos.



b) Mount the specimens in Instron and perform the 90-degree peeling (vertical peeling) to measure the peeling force, as shown in following two photos. The peeling rate was set at 0.5 inch /min.



The typical peeling curves were recorded and shown in following two figures:



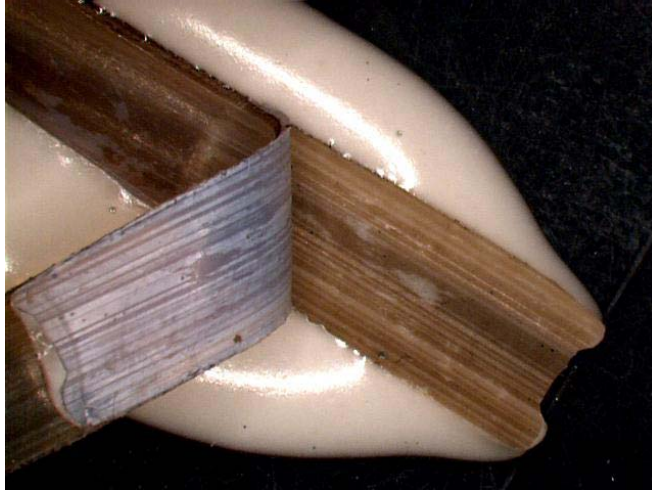
The data in the leveled portion of the peeling curve are averaged as the peeling force. The peeling strength is calculated by normalizing the peeling force to the width of the specimen, which yields a unit of lb / linear inch.

The following Table lists and compares the peeling adhesions and failure modes of the tubing and the wire to adhesives with varied conditions such as chemical treatment times, different adhesives, cure profiles at room temperature or 65°C, and other treatments, etc.

| Parts | Strip Width (inch) | Etch Time (sec) | Adhesive | Cure Condition | Avg Peeling Force (lbf) | Peel Strength (lb/inch) | Failure Mode |
|--------------------|--------------------|-----------------|------------|----------------|-------------------------|-------------------------|--------------|
| P.E. Teflon Tubing | 0.25 | 30 | EP1640 | rmt 24 hrs | 3.2 | 12.8 | Cohesive |
| P.E. Teflon Tubing | 0.19 | 60 | EP1640 | rmt 24 hrs | 3.5 | 18.6 | Cohesive |
| P.E. Teflon Wire | 0.12 | 30 | EP1640 | rmt 24 hrs | 1.8 | 14.7 | Cohesive |
| P.E. Teflon Wire | 0.12 | 60 | EP1640 | rmt 24 hrs | 1.4 | 11.9 | Cohesive |
| P.E. Teflon Tubing | 0.20 | 30 | Supplier X | rmt 24 hrs | 1.3 | 6.5 | Cohesive |
| P.E. Teflon Tubing | 0.14 | 60 | Supplier X | rmt 24 hrs | 1.2 | 8.6 | Cohesive |
| P.E. Teflon Tubing | 0.18 | 30 | EP1640 | 65C 2 hrs | 2.4 | 13.3 | Cohesive |
| P.E. Teflon Tubing | 0.22 | 60 | EP1640 | 65C 2 hrs | 1.5 | 6.8 | Cohesive |
| P.E. Teflon Wire | 0.12 | 30 | EP1640 | 65C 2 hrs | 1.0 | 8.3 | Cohesive |
| P.E. Teflon Wire | 0.12 | 60 | EP1640 | 65C 2 hrs | 1.4 | 11.9 | Cohesive |
| P.E. Teflon Tubing | 0.17 | 30 | Supplier X | 65C 2 hrs | 2.0 | 11.8 | Cohesive |
| P.E. Teflon Tubing | 0.25 | 60 | Supplier X | 65C 2 hrs | 2.5 | 10.0 | Cohesive |
| P.E. Teflon Tubing | 0.22 | 0, No treat | EP1640 | rmt 24 hrs | 0.0 | 0.0 | Adhesive |
| Std PVC Tubing | 0.26 | 0, No treat | EP1640 | rmt 24 hrs | 2.3 | 8.8 | Cohesive |
| P.E. Teflon Tubing | 0.22 | UV 5 min | EP1640 | rmt 24 hrs | 0.0 | 0.0 | Adhesive |
| P.E. Teflon Tubing | 0.23 | Plasma 2 min | EP1640 | rmt 24 hrs | 0.0 | 0.0 | Adhesive |
| P.E. Teflon Tubing | 0.25 | UV 5 min | Supplier X | rmt 24 hrs | 0.0 | 0.0 | Adhesive |
| P.E. Teflon Tubing | 0.22 | Plasma 2 min | Supplier X | rmt 24 hrs | 0.0 | 0.0 | Adhesive |

Some conclusions based on the data listed in this Table have been derived and summarized in the Conclusions section (the last section) of this report.

The peeled interfaces of the specimens were observed under a microscope. As shown in following photo, after the peeling, a dark layer is left on the adhesive side as shown in the photo below. This indicates that the bonding of adhesive to the modified Teflon surface has reached to a cohesive mode. Thus the adhesion is fully optimized.



Conclusions

The wet chemical process developed by United Adhesives is capable to modify the Teflon surface to a hydrophilic nature and improve its adhesion to epoxy adhesive. Experiment shows that a 30 seconds submerging of Teflon surface in wet chemical solution is enough to generate the hydrophilic surface that contains OH and C=O functional groups.

The adhesion of the Teflon to EP1640 epoxy adhesive is significantly improved and fully optimized with the wet chemical treatment. Its peeling strength reaches to a level higher than that of standard PVC tubing plastic. The peeling mode is cohesive along the interface of treated layer with bulk of Teflon.

With the room temperature cure, EP1640 has better adhesion than the competitor's epoxy adhesive, while under 65°C heat cure, both adhesives have compatible adhesion. A longer time submerging of Teflon, such as 60 seconds, did not show significant difference with the adhesion.

Some other surface modification techniques, such as UV ozone, plasma, organic base, have also been applied in this experiment. Results show that they are not as effective as the wet chemical treatment. It needs to emphasize, however, the wet chemical process needs to be handled properly with cautions.