

RTI PAST PERFORMANCE		
RTI Tracking Number:	1311278	Date: 11/15/2013
Core Task:	Metallurgical Testing, Chemical Analysis	
Analytical Techniques	Feature/EDS/FTIR/Photo/SEM/EDS	

Four total samples were received for analysis and were identified as follows:

- 1 – No rubber to plastic failure 1
- 2 – No rubber to plastic failure 2
- 3 – Rubber to plastic insert bond failure 1
- 4 – Rubber to plastic insert bond failure 2

The target of the analysis was to characterize the insert surfaces and bond failure modes using techniques of EDS (Energy Dispersive X-ray Spectroscopy) for qualitative identification of elements with atomic number greater than five, and FTIR (Fourier Transform Infrared Spectroscopy) for identification of organic functional groups. In addition, film build analysis was to be performed on the adhesive interfaces by cross-section optical microscopy. The results of the analysis are discussed below.

Examination of samples 1 and 2 do not show any failure substrate therefore a section was cut from each part to examine the film build at the rubber to inner plastic interface. Samples 3 and 4 were received with catastrophic failure to the insert substrate almost entirely around the circumference with little evidence of rubber tear. The samples as received are provided as Figures 1 and 2. For samples 3 and 4, the insert sections were isolated by completing the rubber tear. Sections were cut from each insert for analysis by EDS, FTIR and film build. The mating rubber backs from samples 3 and 4 were also isolated for examination by EDS and FTIR. In addition, bulk sections of the insert plastic and rubber were analyzed for baseline substrate information.

Examination of the two substrate samples by EDS indicate the dominant presence of elements associated with a Chemlok 6253 adhesive system with no elemental indication of foreign contaminants. For sample 3, however a significant increase in carbon level compared to the other substrate examined indicates more underlying substrate presence (or thinner adhesive in this region). Examination of the two mating rubber backs by EDS indicate the dominant presence of elements associated with a Chemlok 6253 adhesive system with no elemental indication of foreign contaminants.

Examination of the two substrate samples by FTIR indicate molecular absorbance indicative of functional groups of polymeric hydrocarbon, carbonyl, alkoxylation, nitrogen containing and halide containing organics. The same primary absorbance bands were noted at the mating rubber backs, with no indication of molecular absorption due to the underlying substrates. It is of noteworthy mention, however, that for the substrates examined for samples 3 and 4, a significant decrease was noted in the carbonyl band intensity compared to the mating rubber back samples. This is indicative of a discontinuity in composition across the adhesive profile that is not explained by simply the bonding process (as original levels are noted at the mating rubber back in the bonded samples).

Cross-section analysis for dry film thickness at the substrate for samples 1 and 2 indicates a nominal reading of 42.0 microns (1.6mils) for sample 1 and 29.1 microns (1.1mils) for sample 2. Cross-section analysis of the dry film thickness for samples 3 and 4 at an area of slightly retained rubber indicates a nominal reading of 71.9 microns (2.8mils) for sample 3 and 63.0 microns (2.5 mils) for sample 4.



Figure 1 – Samples 1 and 2 No rubber to plastic failure



Samples 3 and 4 – Rubber to plastic bond failure samples

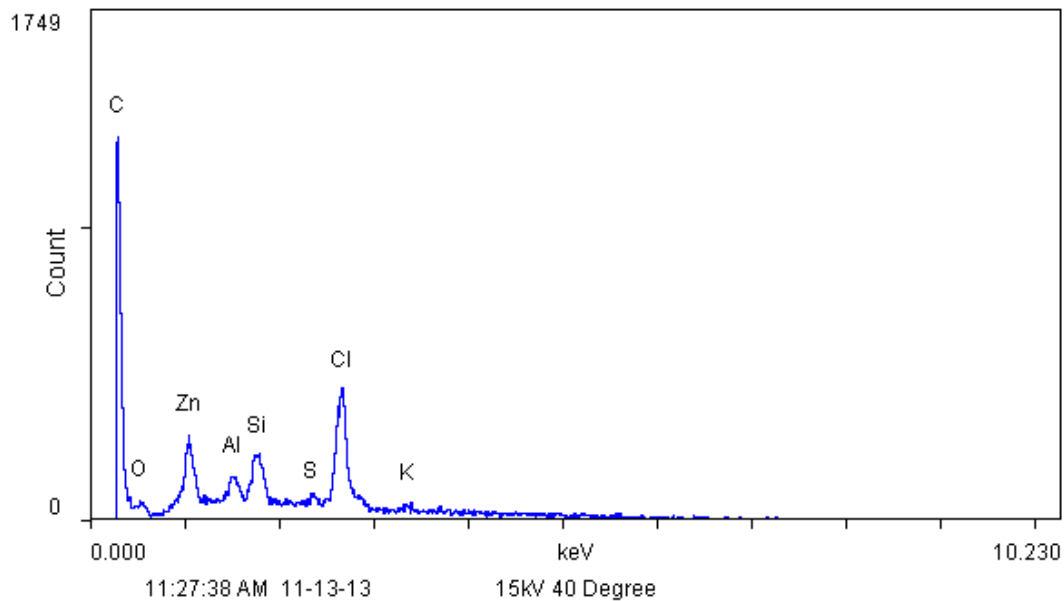


Figure 3 – EDS spectrum obtained from the Sample 3 Failure Substrate - Major elements (>10%) – carbon, oxygen; Moderate elements (2-10%) – chlorine, zinc, silicon; Minor elements (0.2-2%) – sulfur, aluminum, potassium

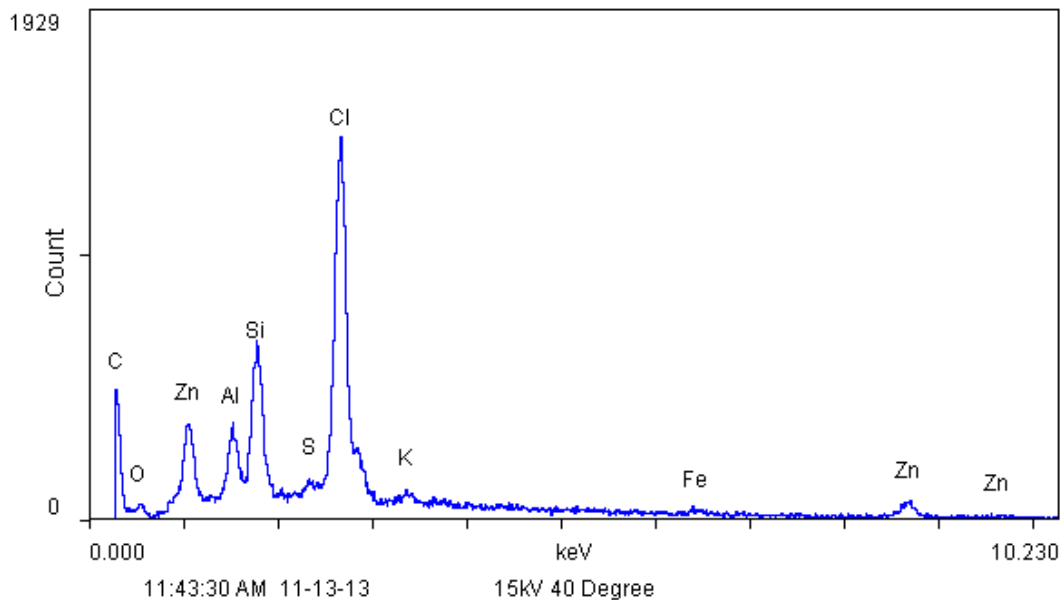


Figure 4 – EDS spectrum obtained from the Sample 3 Mating Rubber back - Major elements (>10%) – chlorine, carbon, oxygen; Moderate elements (2-10%) – zinc, silicon, aluminum; Minor elements (0.2-2%) – potassium, sulfur, iron

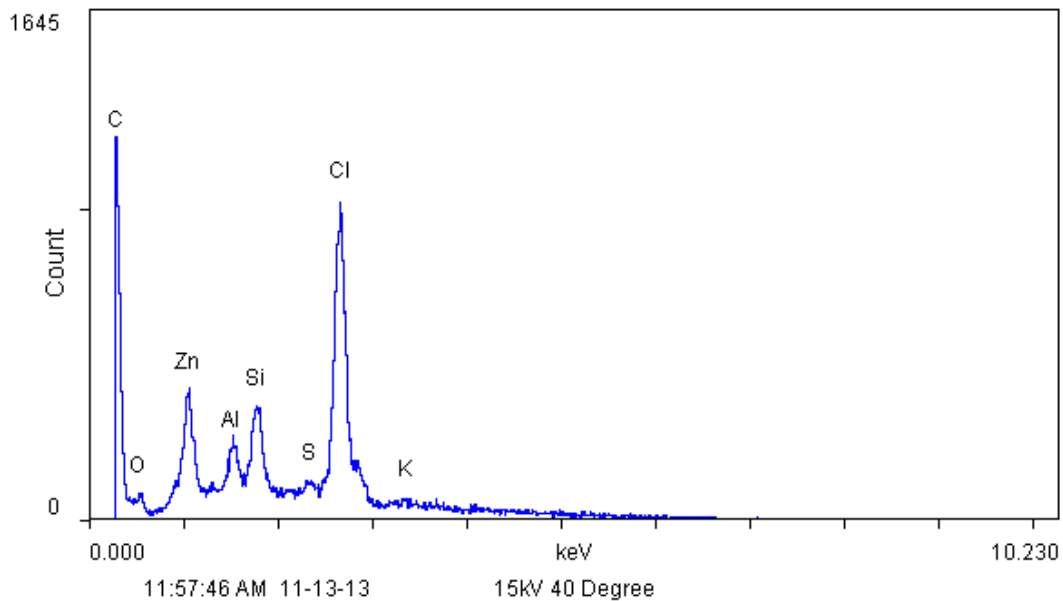


Figure 5 – EDS spectrum obtained from the Sample 4 Failure Substrate - Major elements (>10%) – chlorine, carbon, oxygen; Moderate elements (2-10%) – zinc, silicon; Minor elements (0.2-2%) – potassium, sulfur, aluminum

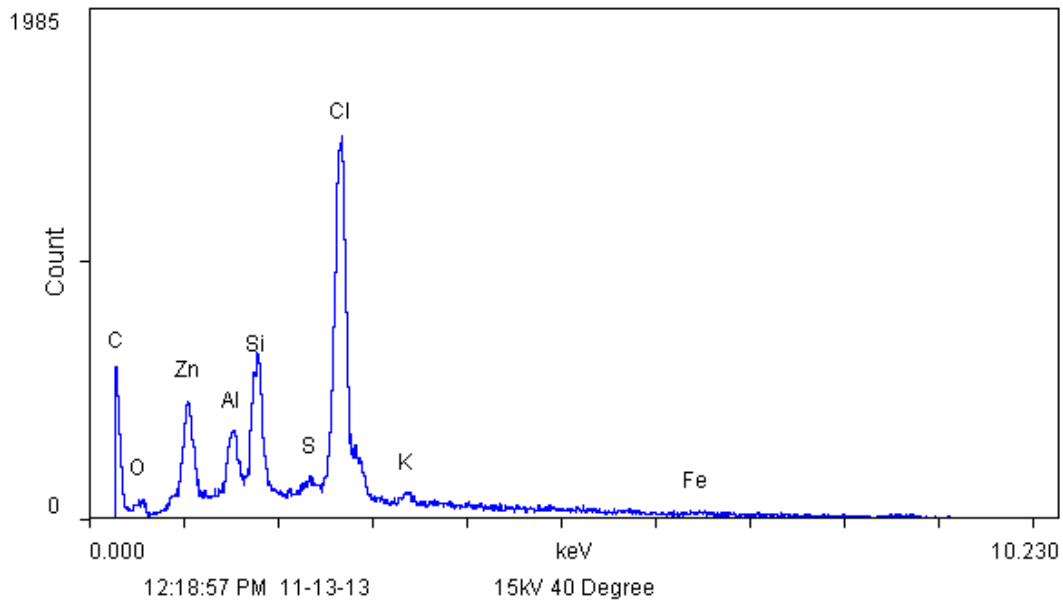


Figure 6 – EDS spectrum obtained from the Sample 4 Mating Rubber Back - Major elements (>10%) – chlorine, carbon, oxygen; Moderate elements (2-10%) – zinc, silicon; Minor elements (0.2-2%) – potassium, sulfur, aluminum, iron

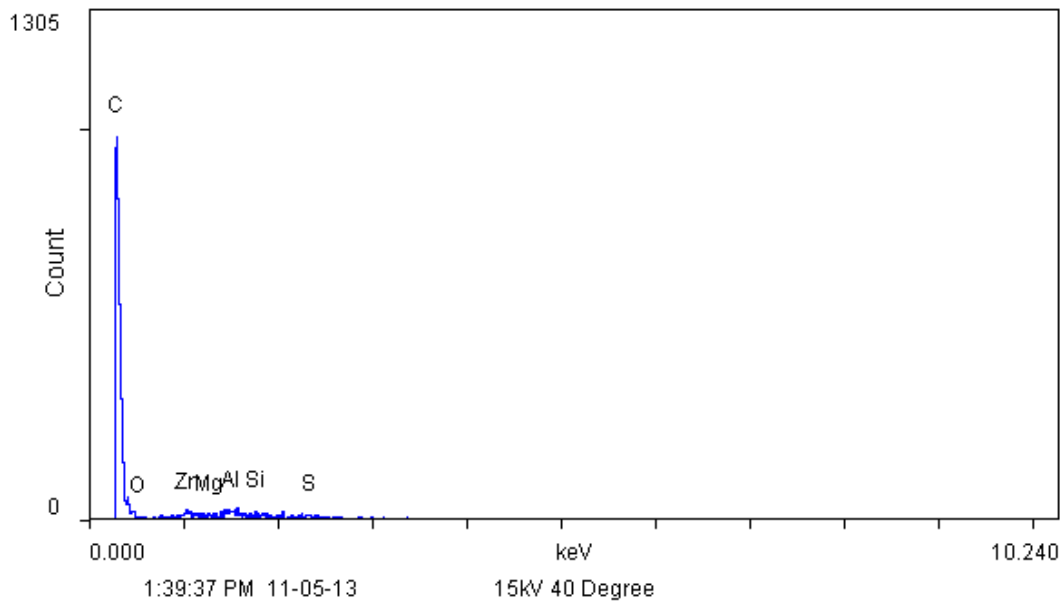


Figure 7 – EDS spectrum obtained from the bulk rubber sample for baseline levels - Major elements (>10%) – carbon; Moderate elements (2-10%) – none; Minor elements (0.2-2%) – zinc, oxygen, magnesium, aluminum, silicon, sulfur

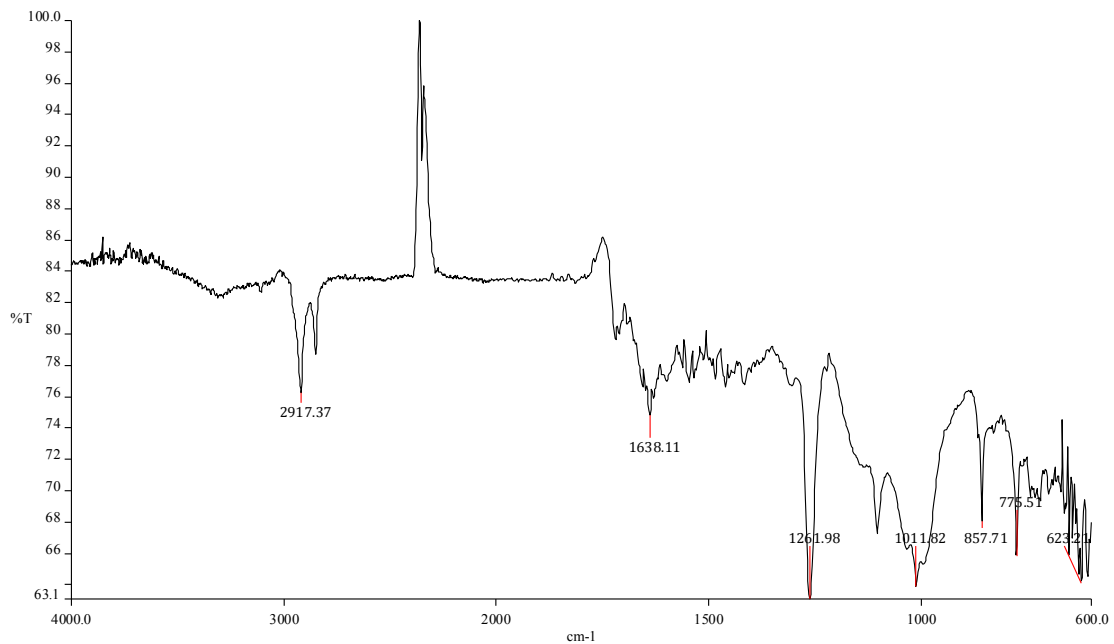


Figure 8 – FTIR spectrum obtained from the Sample 3 Substrate

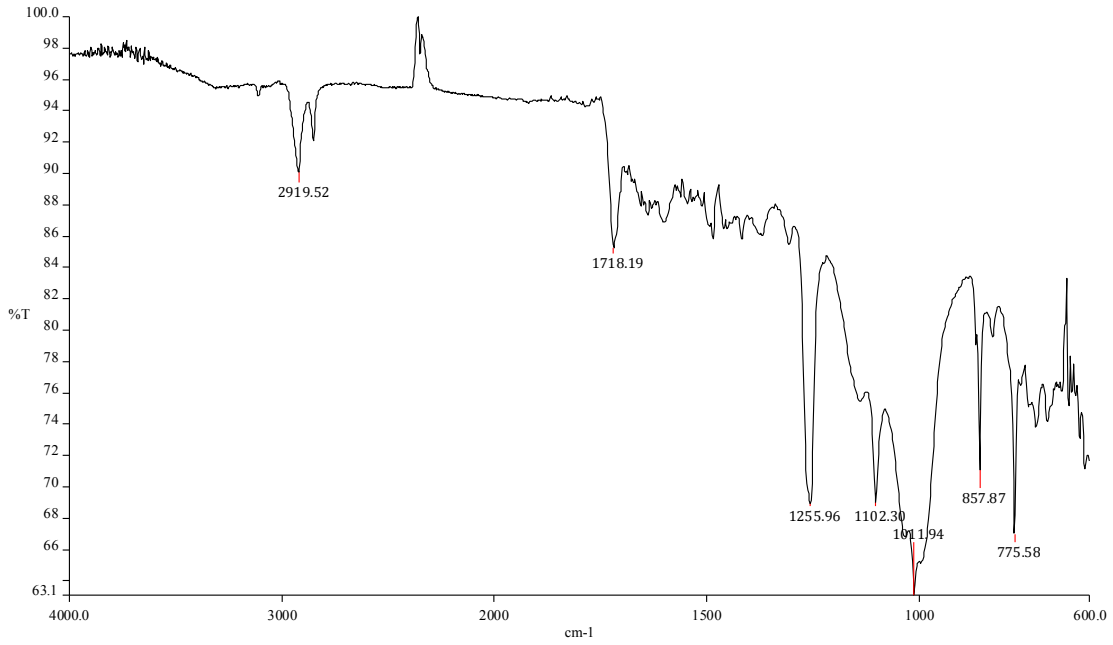


Figure 9 – FTIR spectrum obtained from the Sample 3 Mating rubber back

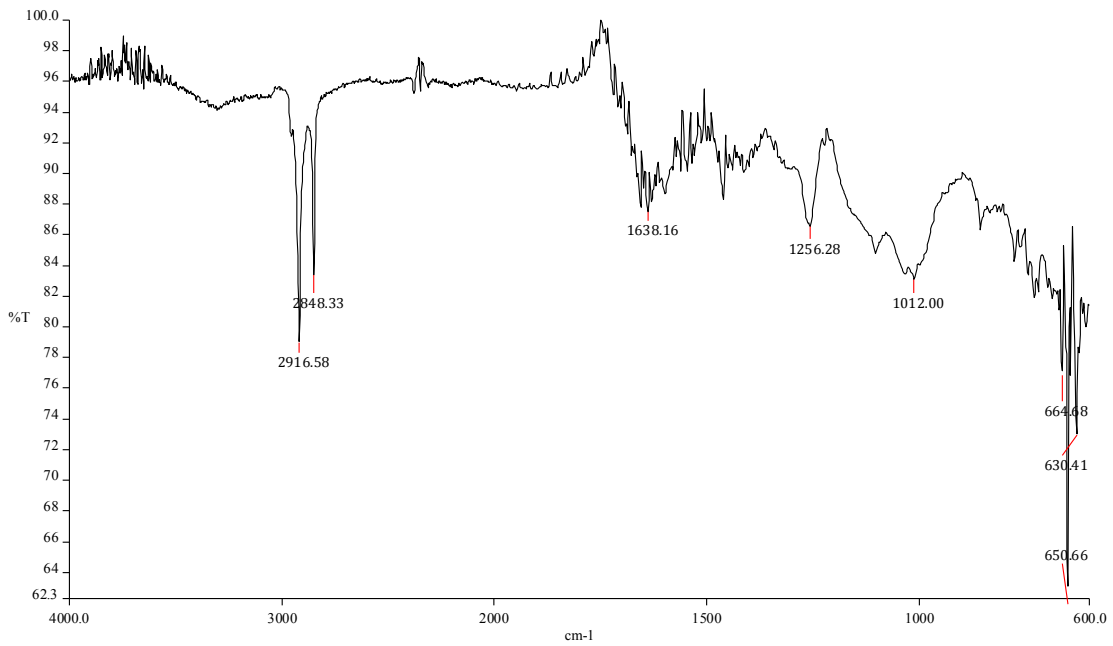


Figure 10 – FTIR spectrum obtained from the Sample 4 Substrate

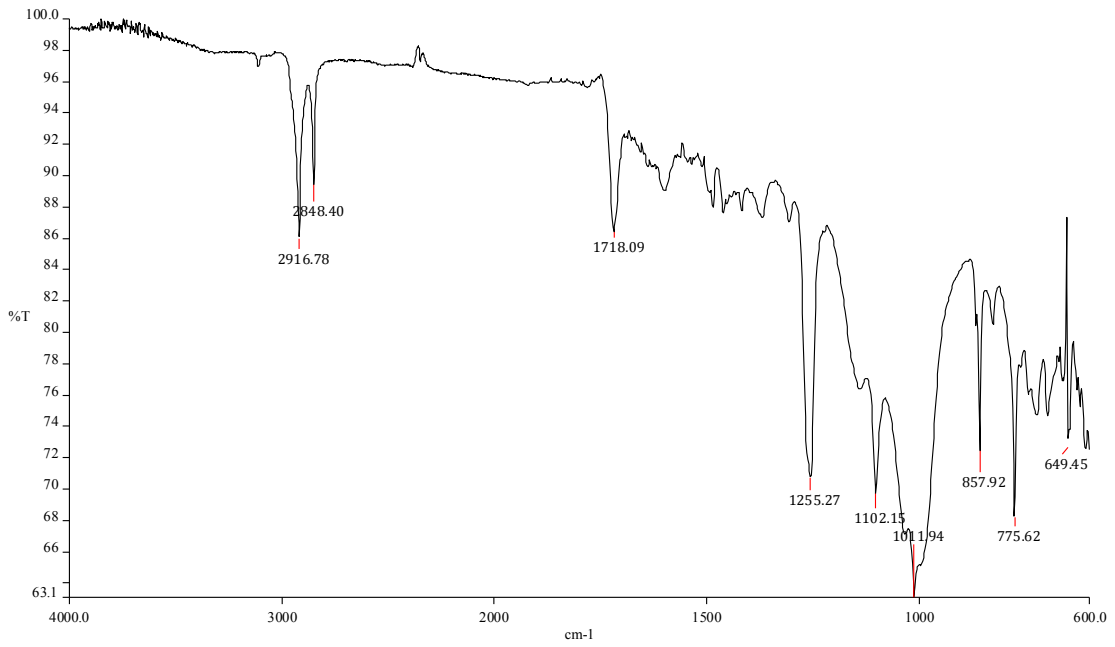


Figure 11 – FTIR spectrum obtained from the Sample 4 Mating rubber back

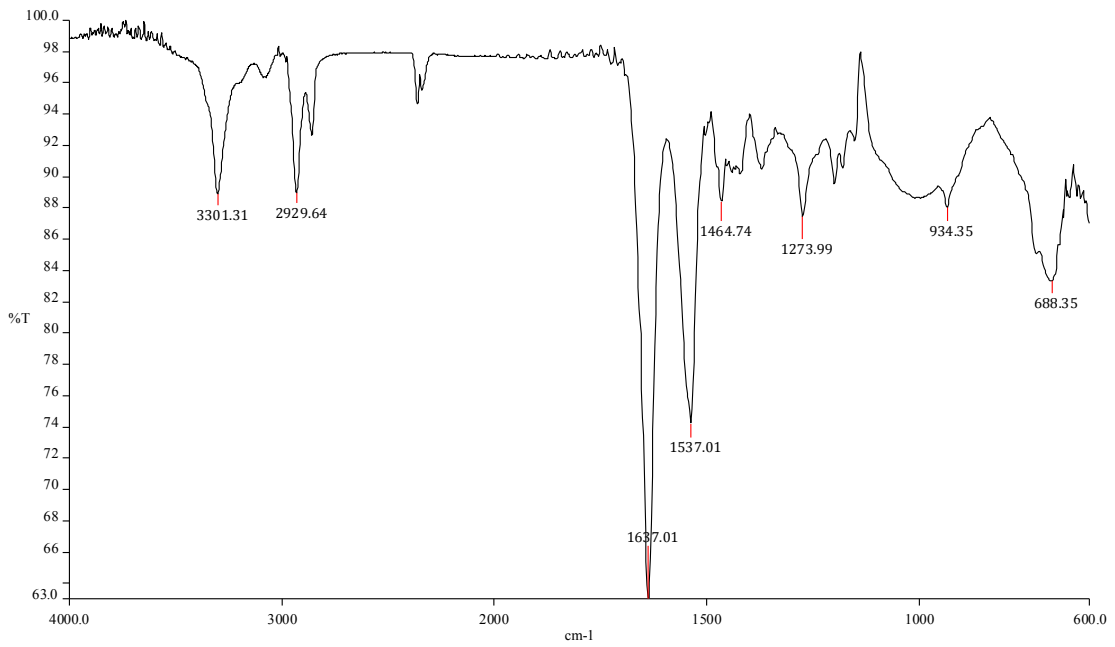


Figure 12 – FTIR spectrum obtained from the bulk plastic

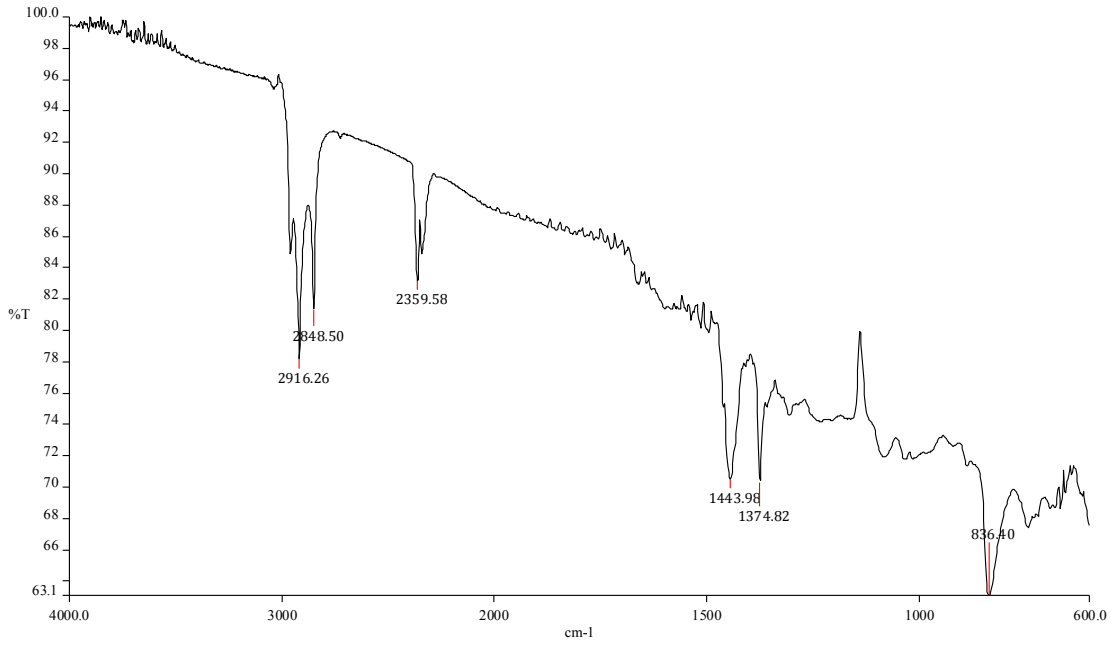


Figure 13 – FTIR spectrum obtained from the bulk rubber

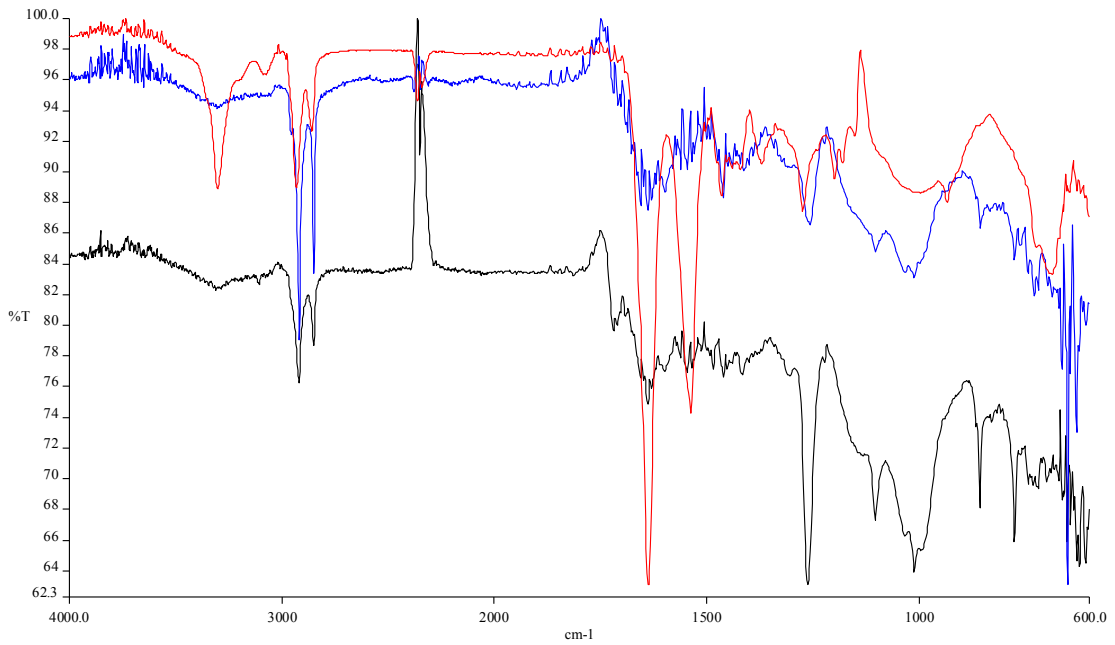


Figure 14 – Overlay of substrate 3, substrate 4, plastic

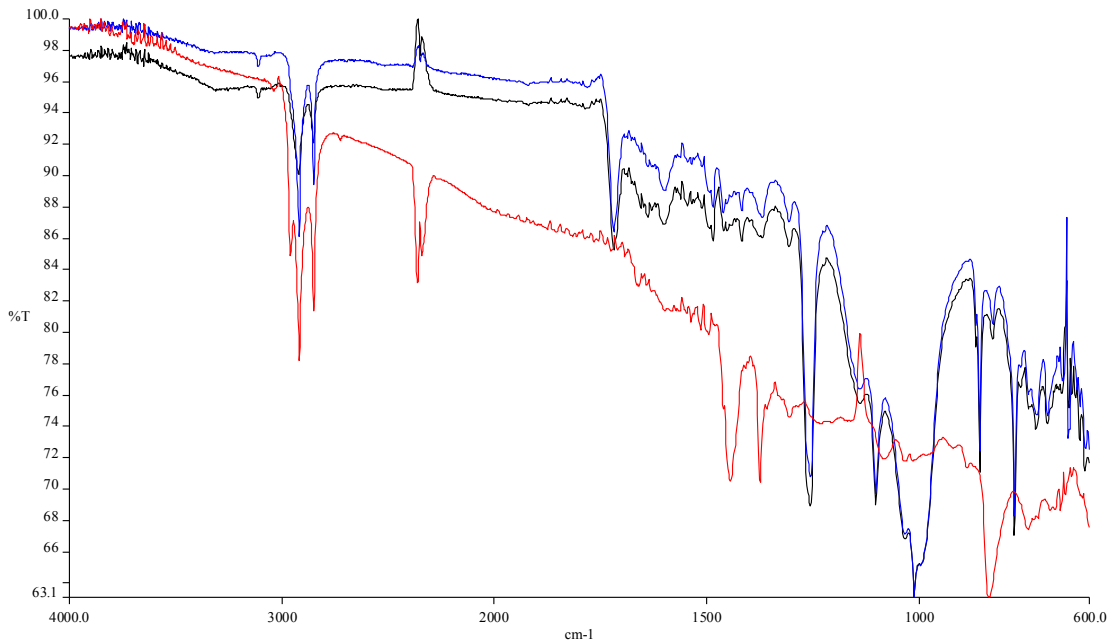


Figure 15 – Overlay of sample 3 rubber, Sample 4 rubber, bulk rubber

Adhesive Thickness:

Analytical Method: ASTM B487-07.

Tested four (4) rubber banded plastic parts were received for analysis. The target of said analysis was to measure the thickness of the adhesive layer by using a variety of metallographic techniques. The results of the analyses follow.

The tested samples with a small amount of banded rubber, which was still attached was mounted transversally, metallographically prepared in accordance with ASTM E3-11 and microscopically examined in the as-polished condition.

The adhesive thickness of the polished surface was measured microscopically.

The measurement was performed at 100x/200x on an Image-Pro Plus Image Analyzer, and reported in microns (μm). Please see the results of the image analysis (Table #1) and micro-image of a representative cross-section and description below.

Table 1.

Description	Layer	Min. Thickness	Max. Thickness	Average
Sample #1	Adhesive	31.85 (μm)	57.79 (μm)	42.04 (μm)
Sample #2	Adhesive	22.96 (μm)	33.34 (μm)	29.14 (μm)
Sample #3	Adhesive	67.60 (μm)	78.57 (μm)	71.94 (μm)
Sample #4	Adhesive	56.30 (μm)	68.89 (μm)	62.96 (μm)

Appendix:

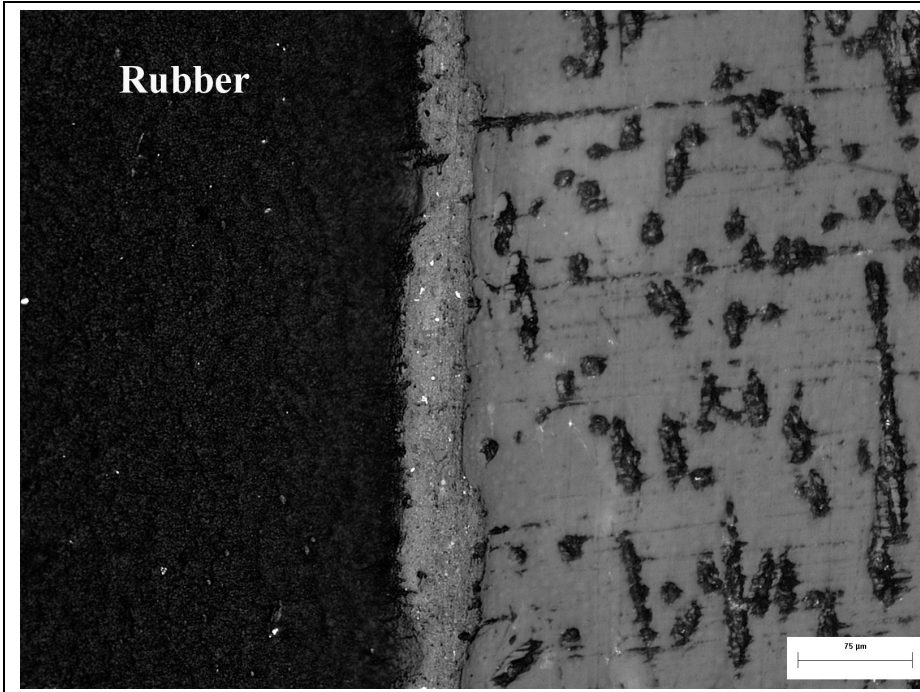


Figure: 16

Sample ID: #1

Magnification: 200X

Condition: As polished

Comments: Micro-image illustrates distribution of the coatings on plastic substrate.

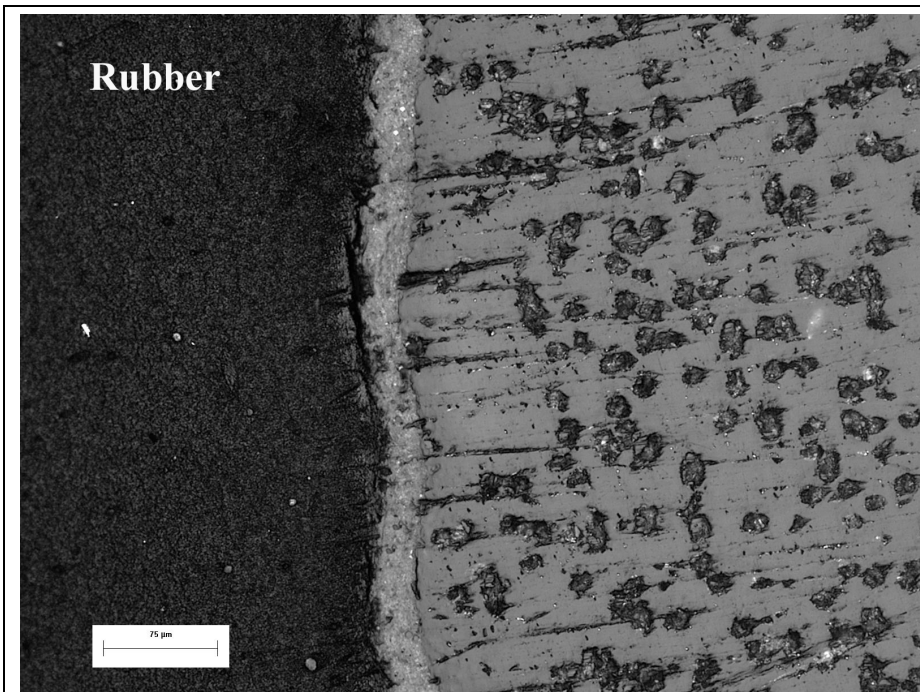


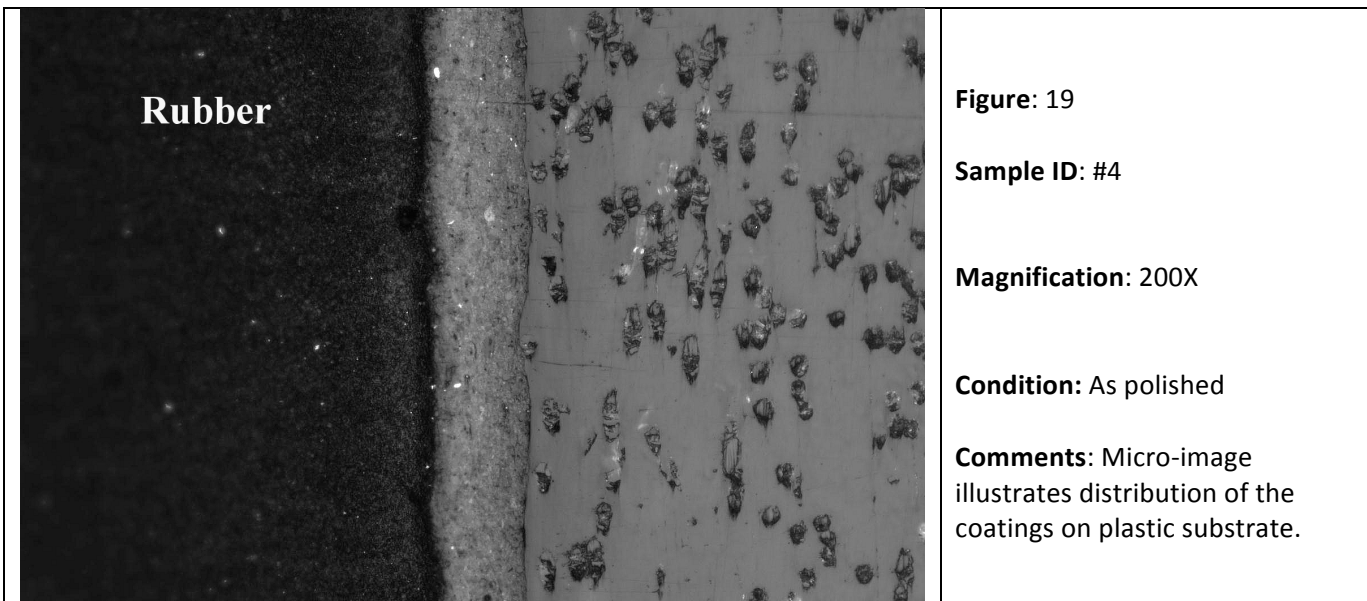
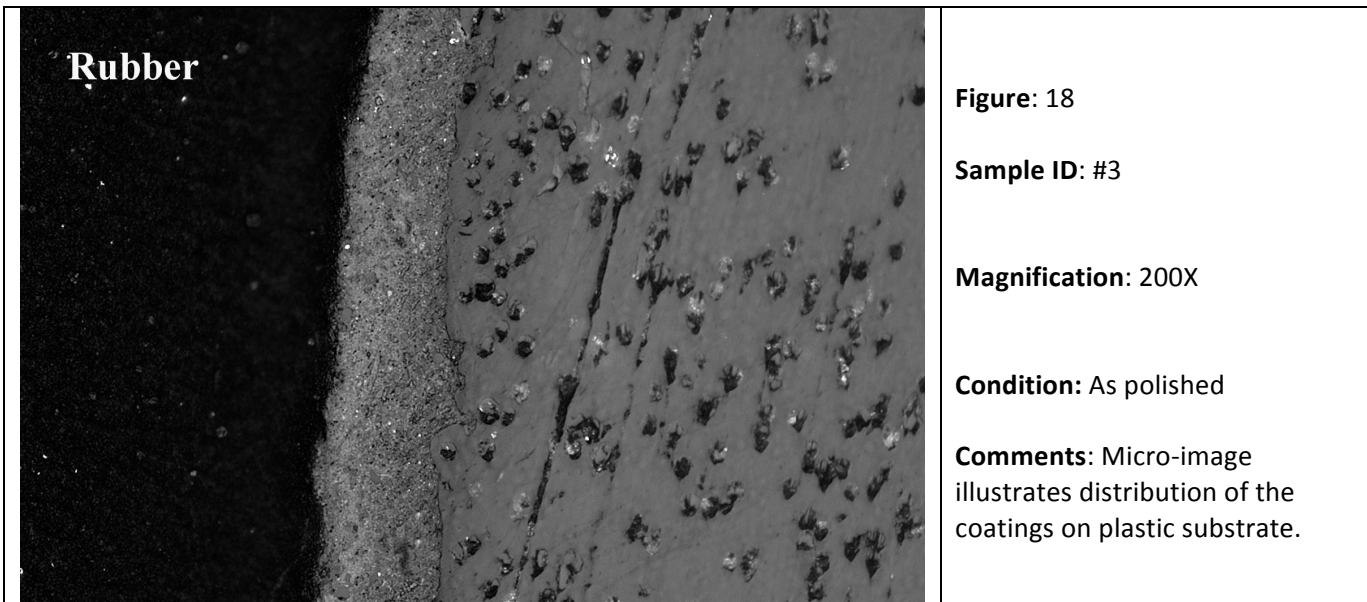
Figure: 17

Sample ID: #2

Magnification: 200X

Condition: As polished

Comments: Micro-image illustrates distribution of the coatings on plastic substrate.

**Conclusion:**

Based on the provided analysis, the dominant failure mode in the failed samples occurred as cohesive within the adhesive system, as significant and contiguous adhesive levels were noted at both mating interfaces for both samples. There is no indication of gross contamination or anomalies within the adhesive system with the exception of a decreased carbonyl level noted at the bonded substrates not evident at mating rubber backs. This does indicate a suspicious phenomenon occurring within the adhesive system or compositional "striations" which can possibly affect the strength of the bonding system. Dry film thickness measurements for samples 1 through 4 indicate film builds within the application guidelines for 6253 system.