ROBUST BONDING THROUGH PROCESS CONTROL

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ABSTRACT

Process control has been proven to be the most reliable means of safeguarding the quality of adhesive bonds according to Federal Aviation Administration (FAA). A method for implementing process control for reduction in risk in a bonded joint fabrication process is demonstrated in this study using a selected bonding system. The stepwise method included risk analysis to identify defects with the highest impact and likelihood to occur, evaluation of various pre-bond surface analysis tools to monitor for the selected defects, and demonstration of the benefits of in-process monitoring utilizing threshold limits determined from bond performance tests. The bonded system selected for investigation was an aerospace carbon fiber epoxy composite substrate surface prepared with random orbital sanding using 180 grit aluminum oxide sand paper. A series of portable, pre-bond surface analysis tools were investigated for their ability to be used for in-line bond process control. Results and threshold limits are presented from roughness, ballistic water contact angle (WCA), color, gloss, and Fourier transform infrared spectroscopy (FTIR) surface analysis tools. Results demonstrated how in-process inspection methods can be used to ensure quality of a surface preparation for a selected bonding system. A framework is provided for implementation of bond process control for robust bonding.

1. INTRODUCTION

The research efforts below are aligned with the recommendations outlined in Federal Aviation Administration’s (FAA) Advisory Circular AC 20-107B [1] which emphasizes verification of repeatable and reliable bonding processing steps utilizing in-line bond process monitoring. Efforts are also ongoing within the FAA Joint Advanced Materials Structures Center of Excellence (JAMS) consortium to further develop and define a system for bond process control [2].

This work further investigates process control methods to demonstrate the benefits of in-line bond process monitoring. A significant amount of work has been done in the past outlining an efficient primary structure certification method including robust process control [3]. Some of this work led to a system for monitoring and verifying a bond process that optically monitors the bonding workstation [4]. The study demonstrated a method to implement process monitoring on a selected bonded system utilizing quantitative inputs from pre-bond surface analysis tools.

1.1. Approach for Bond Process Control Implementation

The step wise process for implementing a process control system for bonded joint fabrication demonstrated here is outlined below:

1. Bonded Joint Definition

2. Risk Analysis and Identification of High Risk Process Parameters
3. Evaluation and Selection of Tools to Monitor High Risk Parameters
4. Integration of Monitoring Tool Outputs into a Process Control System

1.2. Bonded Joint Definition

The first step in development of robust bond process control is a full assessment of the bonded joint materials and processes that contribute to the performance of the bond. For this work, the process selected was sanding of an epoxy composite substrate followed by solvent wiping prior to bonding as outlined in Figure 1. Manual sanding was selected because it is a highly variable process with a significant number of factors that can affect bond performance. Several parameters control the level of sanding including pressure, disk speed (revolutions per minute, RPM), number of passes and overall time of sanding. An attempt was made to document any process parameters or factors that may impact bond performance.

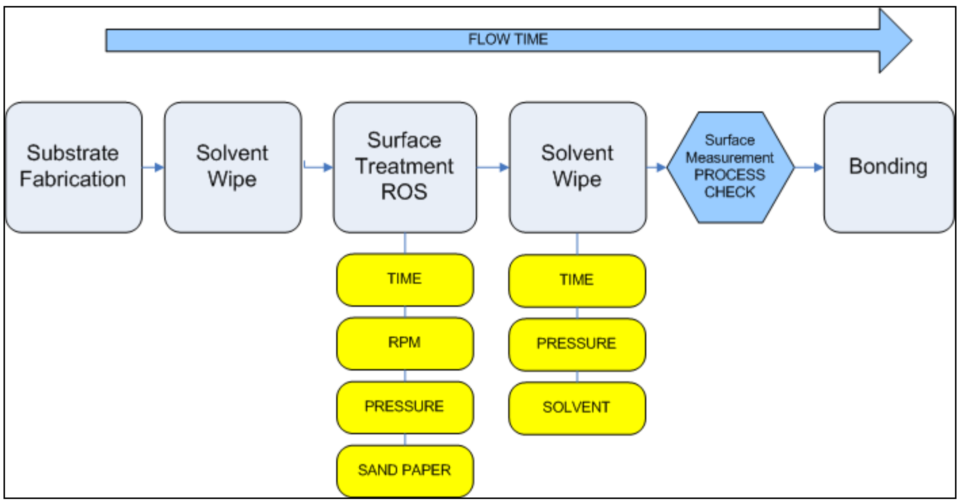

Figure 1. Bonded joint materials and process definition.

1.3. Risk Analysis and Identification of High Risk Parameters

The purpose of performing a risk assessment is identification of high risk process parameters that should be selected for monitoring and process control. Several methods are available for risk assessment in the industry including Bayesian analysis [5] and fault tree analysis. In each of the risk assessment methods, the results are based on input from subject matter experts which should be reassessed periodically as the process develops. In this test case the risk analysis system selected for evaluation of the bonded system was the Likelihood – Consequence Risk Assessment method.

The next step involved identifying all the risks associated for each process step or materials is shown in Table 1. Next, each risk was assessed for likelihood of happening and the consequence if the event occurs.

Table 1. Likelihood-Consequence Risk Assessment of Composite Surface Preparation with Sanding.

ID	IDENTIFIED RISKS [Risks in BOLD CAPS were selected for investigation.]	Likelihood 1 - unlikely 5- highly likely	Consequence 1 - no impact to bond performance 5 - known bond failure	Risk Score BEFORE Mitigation	NEW Risk Score AFTER Mitigation (Likelihood = 1)
MATERIALS					
1	No gloves / hands have no contaminant / Natural oils from hands gets on part	2	2	LOW	LOW
2	No gloves / hands have contaminant	2	3	LOW	LOW
3	Wrong gloves - silicone residue	2	3	LOW	LOW
4	Wrong solvent	3	1	LOW	LOW
5	Contaminated solvent	1	3	LOW	LOW
6	Wrong wiper	2	1	LOW	LOW
7	Wrong grit - too large, rougher/pitted or damaged surface	3	1	LOW	LOW
8	Wrong grit - too small, not aggressive enough	3	1	LOW	LOW
PRE ABRADE SOLVENT WIPE (Pre Sand Surface) - Risks captured and investigated with "No Sanding" [Risk 9]					
NA	No solvent cleaning - skip solvent wipe entirely	2	1	LOW	LOW
NA	Dry wipe only - no solvent on wiper	2	1	LOW	LOW
NA	No dry wipe after solvent wipe - solvent and residue left to dry on surface	2	1	LOW	LOW
ABRADE WITH RANDOM ORBITAL SANDER					
9	**NO SANDING - SKIPPED**	2	5	MEDIUM	MEDIUM
10	Wrong sand paper - too rough - (for example 180 micron / 100 grit)	3	2	MEDIUM	LOW
11	Right sand paper - too aggressive sanding - black dust - fiber damage, sanding for too long	3	2	MEDIUM	LOW
12	Wrong sand paper - too light - 300 grit sand paper, nominal sanding process [Risk captured by Risk 14]	2	4	MEDIUM	LOW
13	Wrong sand paper - 300 grit with longer time get reduced gloss	2	2	LOW	LOW
14	**TOO LIGHT SANDING, LESS TIME, LESS PRESSURE (Correct Sand Paper)**	3	5	HIGH	MEDIUM
15	Sand paper not changed between panels for several weeks. Grit reduced (too light). Risk investigated with [Risk 14]	1	5	MEDIUM	MEDIUM
16	Sand paper not changed on same panel. Grit reduced (too light). Risk investigated with [Risk 14]	4	4	HIGH	LOW
17	Sand paper not changed. Adhesive from sand paper transferred to substrate. Transfer of contaminant from one panel to another.	3	3	MEDIUM	LOW
SOLVENT WIPE (Post Abrade of Surface)					
18	No solvent cleaning (no dry wipe) Sanded dust left in place.	2	3	LOW	LOW
19	Contaminated air - mitigated by solvent wiping and mirror check.	2	2	LOW	LOW
20	Dry wipe only - No solvent on wiper.	2	2	LOW	LOW
21	Contaminated wiper - reused / contaminated.	2	2	LOW	LOW
22	No dry wipe after solvent wipe - solvent and residue left to dry on surface	3	2	MEDIUM	LOW
CUMULATIVE DEFECTS					
23	[Risk 17 - Sand paper not changed.] + no solvent wipe.	2	3	LOW	LOW
24	No solvent wipe + [Risk 9 – No Sanding] + no solvent wipe	1	3	LOW	LOW

Results from the Likelihood-Consequence Risk Assessment were charted on a matrix below (Figure 2). The *quantity* of risks are shown before and after mitigation. The mitigation step here is process control. It is assumed that likelihood of the risks occurring will reduce to 1 with process control.

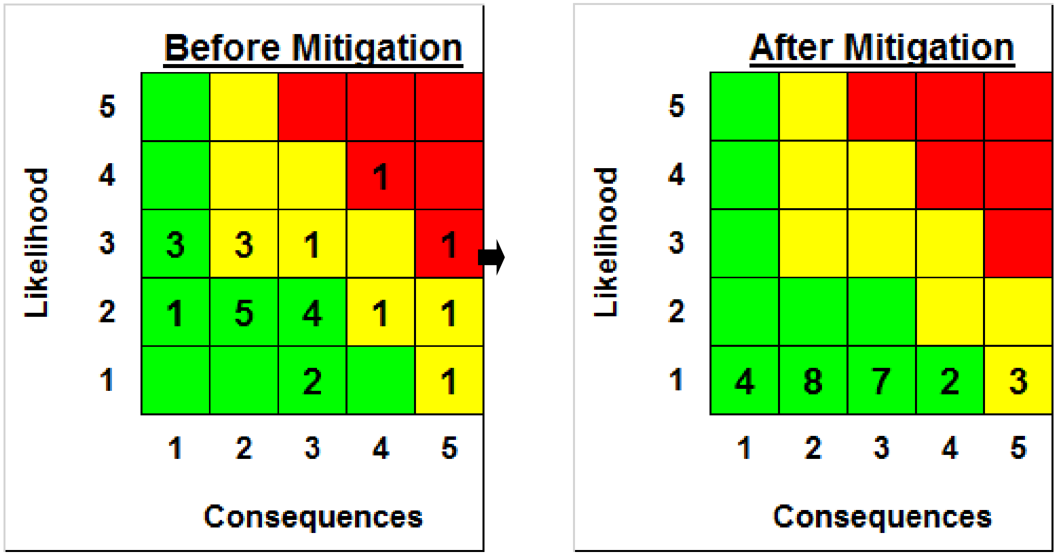

Figure 2. Likelihood-Consequence Risk Analysis before and after Mitigation with Process Control.

The outcome of the Risk Assessment resulted in identification of three "medium" risks with a consequence of five shown in the right hand chart of Figure 2. The three risks are all related to the sanding surface preparation operation:

- Risk 9. No Sanding. Skipped.
- Risk 14. Too light sanding, less time, less pressure.
- Risk 15. Sand paper not changed between panels for several weeks. Grit reduced (too light). Captured under risk 14.

Only Risk 9 and Risk 14 were used as variables for further investigation because Risk 15, "grit reduced", was considered to be captured under "too light sanding", Risk 14.

1.4. Evaluation and Selection of Methods to Monitor High Risk Parameters

The next step in the assessment was to identify methods to measure the surface preparation process *quantitatively*. Various analytical tools, including roughness, ballistic water contact angle (WCA), color, gloss, and Fourier Transform Infrared spectroscopy (FTIR), were evaluated for their ability to assess presence and level of surface preparation / sanding. In addition to analytical measurement, process parameters such as sanding time, pressure and equipment settings were also evaluated. Both the analytical tool results and the process parameter variables were intended to be integrated into the process control system or enhanced bonding workstation described above. A more tightly controlled process narrows the limits in which an operation can take place, and improves the reliability of the bonding process resulting in a more robust end product of a bonded joint. An additional benefit of the in-line process control with video monitoring is a digital record enabling downstream trouble shooting if an issue arises in the field on a specific bonded part.

1.5. Pre-bond Surface Analysis

Surface analysis tools were selected based on their potential to detect the surface preparation, their rapid measurement capabilities and ability of their output to be used as a "go/no go" check in a real time process control system. The goal is for the tool to be utilized as an inline production check to verify if a bonding process step has occurred and been done correctly. For this study, six different surface analysis tools were investigated.

1.6. Flow Time

Tracking process flow time is another method of assessing bond process reliability and consistency. If a process step falls outside the normal, known flow time, it is flagged. Example of an output from a bond process monitoring system is shown in Figure 3.

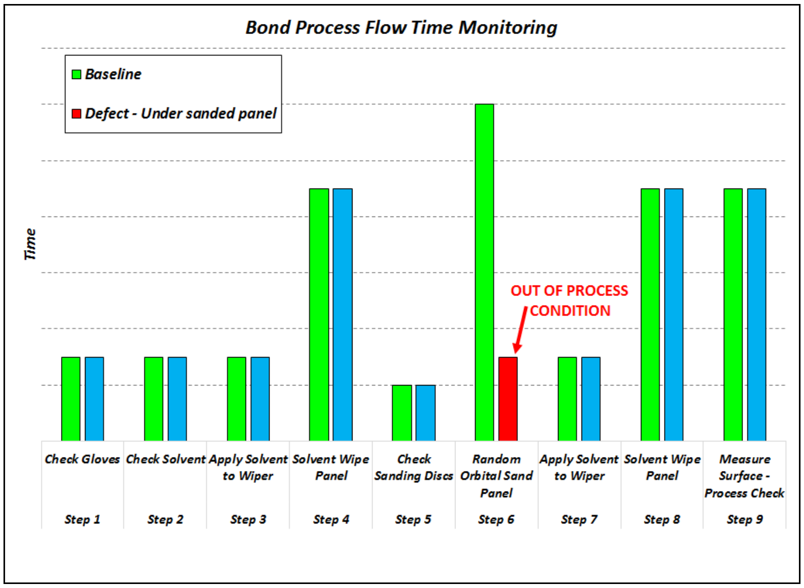

Figure 3. Example of Bonding Flow Time Tracking Defect Identification.

1.7. Integration of Monitoring Tool Outputs into a Process Control System

The end goal of the stepwise bond process control development is its implementation for production of bonded parts. An example of a graphic user interface for implementation in the shop is an enhanced bonding work station shown below in Figure 4. The functionality of the system includes process flow time monitoring, documentation of the process steps as well as in-process checks. This study focuses on definition of the quantitative outputs from the analytical tool outputs for integration into the system as "go/no go" process checks. Future work will incorporate this functionality into the system in order to set and control the operational limits. The end goal of defined and controlled limits will be better in-line bond process control system and the ability to produce robust bonds consistently and reliably.

Figure 4. Optically Enhanced Bonding Workstation for Bond Process Control.

2. EXPERIMENTATION

2.1. Composite Panel Fabrication

Composite substrates were fabricated using 10 plies of 177 ºC (350 ºF) cure carbon fiber epoxy prepreg. The eight inner plies were unidirectional tape (Torayca P2352W-19 T800S/3900-2B UD) and the two outer plies were fabric (Torayca FM6673G-37K T830H-6K-PW/3900-2D). Panels were cured against a tool treated with Frekote 710NC mold release agent. Panels were solvent wiped prior to and after sanding with Eastman™ methyl propyl ketone (MPK) - methyl isobutyl ketone (MIBK) mixture [6] using cleaning cloths, meeting the requirements of AMS3819B Class 2 Grade A [7].

2.2. Surface Treatment

Panels were surface treated by manually sanding with a random orbital sander (ROS) and 180 grit aluminum oxide Merit sand paper disks for various times (Figure 5). For this study, time was used as the variable.

For assessment of FTIR and Optically Stimulated Electron Emission (OSEE) analytical tools, a ladder panel (Figure 6) with various levels of sanding was used. For assessment of all other surface analysis tools, an array of panels were utilized and sanded for 0, 5-10, 10-20, 30 and 60 seconds. Sanding for 1 minute was considered to be the baseline.

Figure 5. Manual ROS sanding of epoxy composite panel.

Figure 6. Sanded ladder panel.

2.3. Surface Analysis

Pre-bond surfaces were characterized before and after surface treatment. Surfaces were evaluated by measuring roughness, WCA, color, gloss, and FTIR chemical signature information. A Keyence VHX-2000 (Version 2.3.5.1) multi-scan digital microscope was used to image the surface. A polarizer and glare reduction setting was used to accentuate surface morphology. Roughness (R_a and R_z) was measured using a Fowler portable roughness tester Model 54-410-500. Ballistic drop deposition, WCA was measured with a Surface Analyst Model SA1001 from BTG Labs. Color was measured with a BYK Gardner spectro-guide 45/0 gloss Model CC-6801 using a Commission Internationale de l'Elcairage (CIE) Lab color scale. Gloss values at 20, 60 and 85 degree illumination angle geometries were also collected using a BYK Gardner micro-TRI-gloss micro Model 4435 instrument. Chemical signature information was gathered using FTIR spectroscopy with an Agilent Model 4100 "Exoscan" spectrometer, gain of 243, 64 scan, 8 cm^{-1} wavenumber resolution between 650 and 4000 wavenumbers and a diffuse reflectance attachment. Optically Stimulated Electron Emission (OSEE (Figure 7) was performed at Boeing with an instrument developed by NASA [8] using an ultraviolet (UV) lamp set point of 3041, grid offset of -41 and peak to peak amplitude of 3.7.

Figure 7. Measurement of sanded composite surface with OSEE instrument.

3. RESULTS

A summary of the results of the various surface analysis measurements collected are shown below in Table 2.

Table 2. Surface Analysis Methods for In-Line Process Control of Composite Sanding Step

Surface Analysis	**Ability to detect level of surface preparation**	
Digital Microscopy	-	no
Roughness with Stylus, R_a, R_z	o	differentiated between sanded and no sanding
Ballistic Water Contact Angle	-	no
Color - a* and b* color values	-	no
Color - L* and E* color values	o	differentiated between sanded and no sanding
Color - delta L* and delta E*, indiv max color values	+	yes
Gloss - 20, 60, 85 deg	o	differentiated between sanded and no sanding
Gloss - 85 deg, indiv max	+	yes
FTIR	+	yes, high standard deviation
OSEE	+	yes, reaches threshold limit

The success of these surface analysis methods is specific to this bonded system: random orbital sanding with 180 grit aluminum oxide of carbon fiber epoxy composite fabric surface. All of the techniques shown can provide guidance for potential usage on other substrates and surface preparations. However, for the purposes of this investigation, results were specific to this bonded system only. Results identified four different analytical tools that can be used to set limits for composite surface preparation with manual ROS sanding. These tools define the operating window and narrows the limits on sanding parameters. Utilization of these tools enables better process control resulting in robust and reliable bonding.

3.1. Microscopy

Digital microscope images of the unsanded and sanded surfaces at 200X are shown in Figure 8. Scratches were observed even on the non-sanded surface indicating that they were likely from solvent wiping or panel handling. A depth analysis (Figure 9) of the 1 minute sanded baseline panel confirmed that the sanding surface preparation step generated a smoothed out surface with no detectable troughs or valleys. This lack of roughness was potentially why the water contact angle wettability method and roughness measurements methods were not good indicators of surface preparation levels.

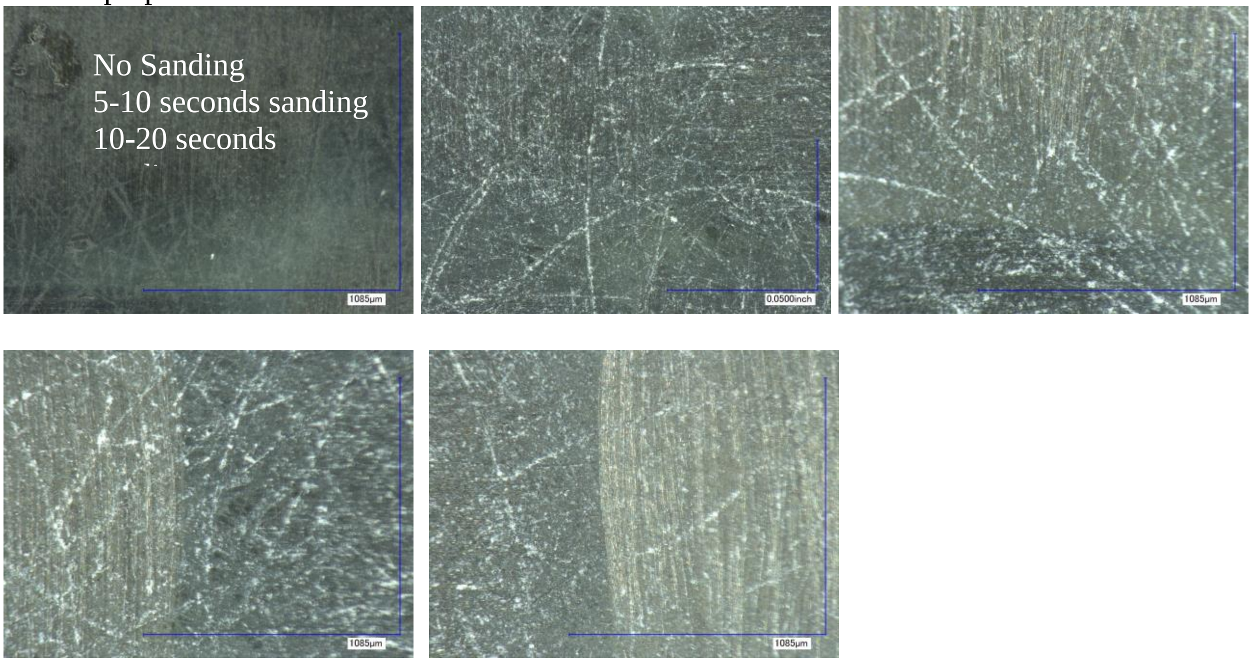

Figure 8. Microscopy images of epoxy composite sanded surfaces, 200X, polarizing filter.

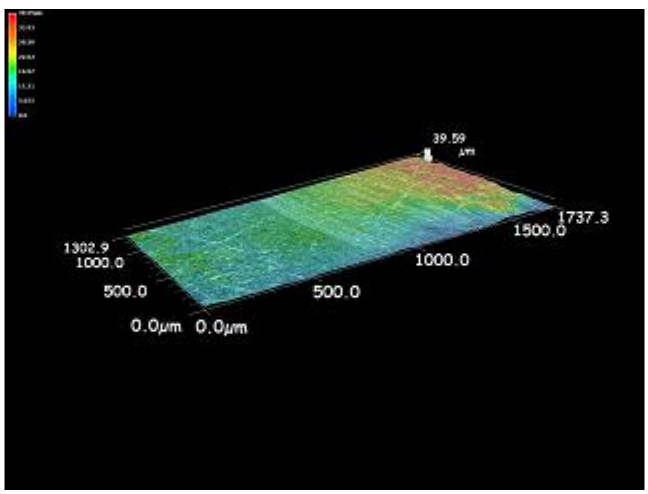

Figure 9. Surface roughness depth analysis of sanded composite, 1 min, 180 grit Al Oxide.

3.2. Roughness

Roughness measurements, R_z and R_a, of sanded composite surfaces are shown in Figure 10. Roughness measurements were able to detect whether a composite surface had been sanded, particularly R_z. However, roughness was not a good indicator of level of sanding. This was partially due to the high level of variability in the roughness values. Previous studies have shown that roughness measured with a profilometer was significantly different between untreated and grit blasted surface preparations on BMI composites [9]. However, measurement of roughness with a stylus type instrument was not successful on epoxy composite surfaces sanded with 180 grit sandpaper using a ROS. Further investigation is needed into other in-line quality control tools beyond the stylus profilometer to quantify the roughness of the hand sanded surfaces.

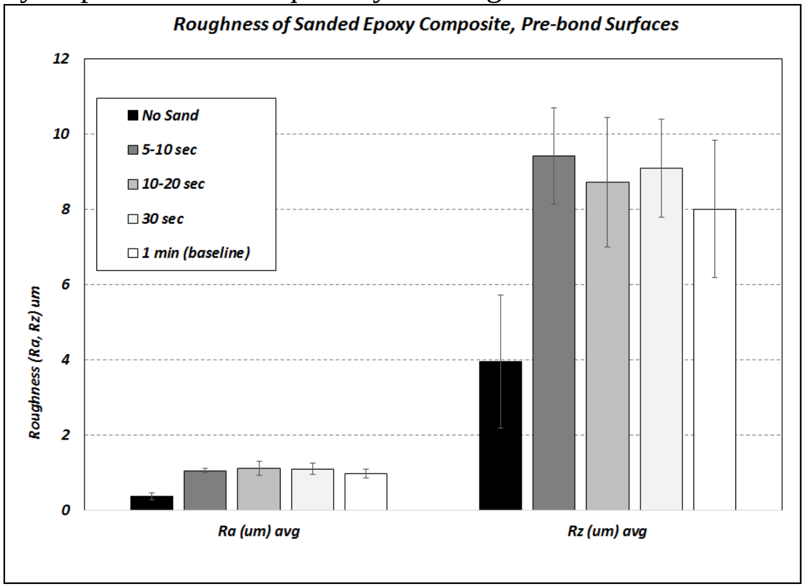

Figure 10. Detection of sanding using Roughness, R_a, R_z.

3.3. Water Contact Angle (WCA)

WCA was not a good indication of sanding surface treatment as there was insufficient variation in surface energy with sanding time to be useful, as shown in Figure 11. There was the option of generating a roughness factor and multiplying the contact angle by this value to adjust for roughness [10]. This would require significant preliminary work to generate a factor for each level of roughness and sanding level making it a less attractive for in-process control.

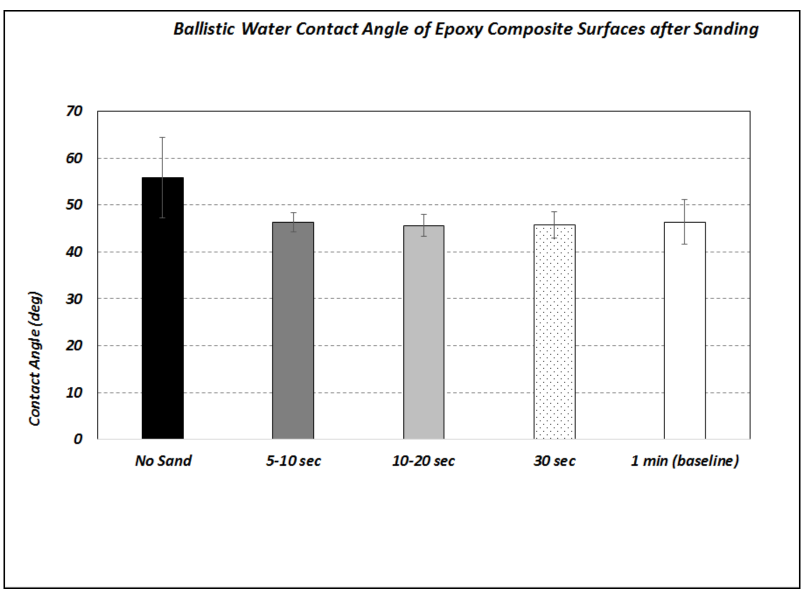

Figure 11. Limited detection of sanding using ballistic water contact angle.

3.4. Gloss

Gloss was investigated as a way to quantitatively assess the level of sanding. Gloss measurements of sanded surfaces, collected at 20, 60 and 85 degrees, are shown in Figure 12. Results showed that gloss measurements could detect whether a surface had been sanded, but not the level. Gloss at 85 degrees was the recommended geometry for low gloss, matte surfaces. As a result, individual maximums of 85 degree gloss did show correlation to sanding levels (Figure 13).

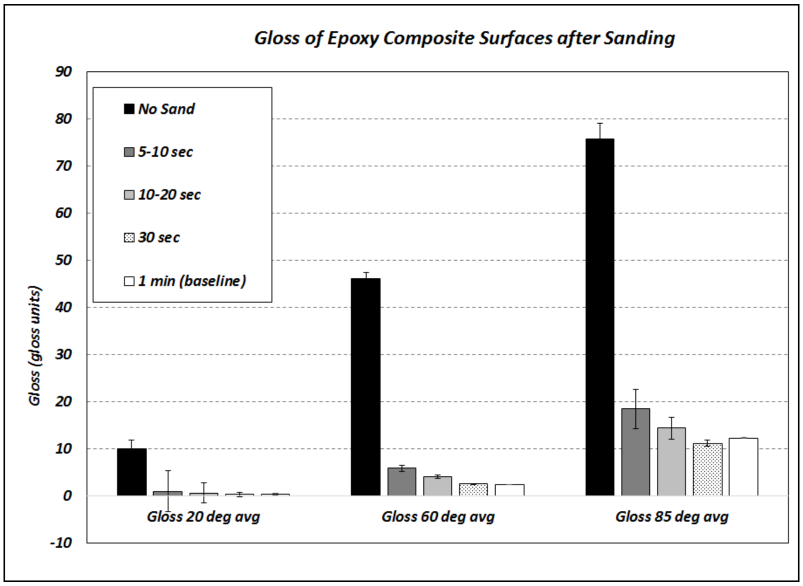

Figure 12. Detection of sanding using gloss, 20, 60 and 85 degree.

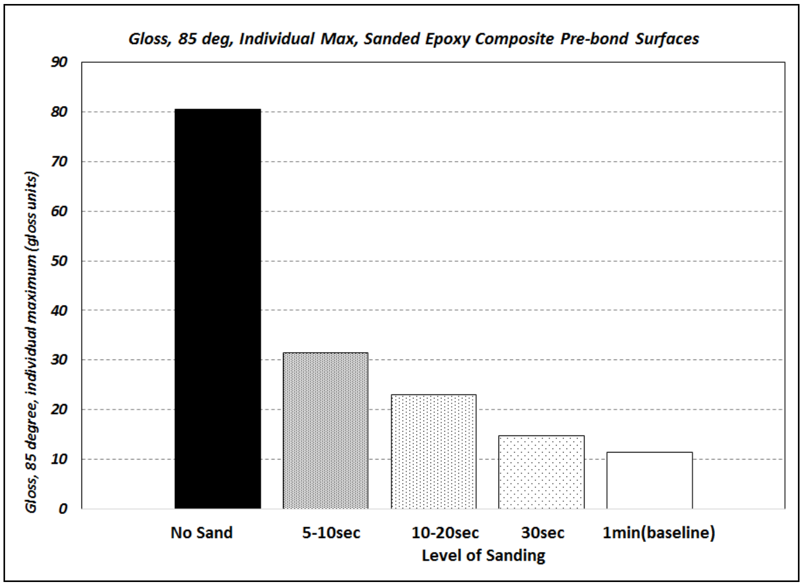

Figure 13. Detection of <u>levels</u> of sanding using gloss, 85 degree, individual maximum.

3.5. Color

Color results are presented in Figure 14. Sanded and unsanded surfaces were distinguished using L* values. However, the direct color measurement did not detect level of sanding. When ΔE* and ΔL* values were calculated, as a difference from the baseline control, and the individual maximum value collected, there was an obvious correlation to level of sanding (Figure 15). Both ΔE* and ΔL* individual maximums, were good candidates to be used as a quality control tool for in-line bond process monitoring with this bonding methodology.

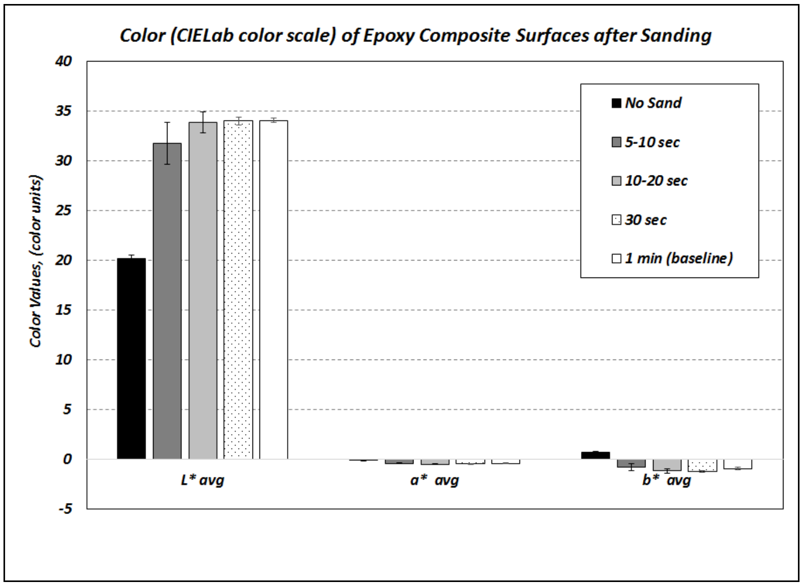

Figure 14. Detection of sanding using Color values, L*a* and b*

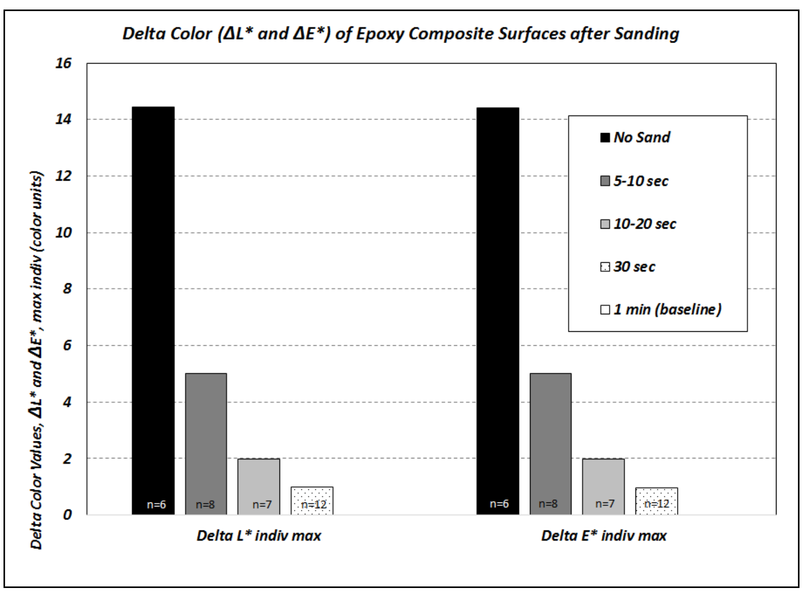

Figure 15. Detection of <u>level</u> of sanding using Delta Color values, ΔE* and ΔL*.

3.6. FTIR (Fourier Transform Infrared Spectroscopy)

Overall FTIR spectra from composite panels with various levels of sanding are shown in Figure 16. Spectra are shown on a common scale. A decrease in the overall FTIR signal was observed with increased sanding potentially due to the reduction in organic epoxy resin on the surface with increased sanding time. Peak area analysis was performed in the region between 3016-2785 cm^{-1} representing the C-H bonding region of the epoxy polymer (Figure 17).

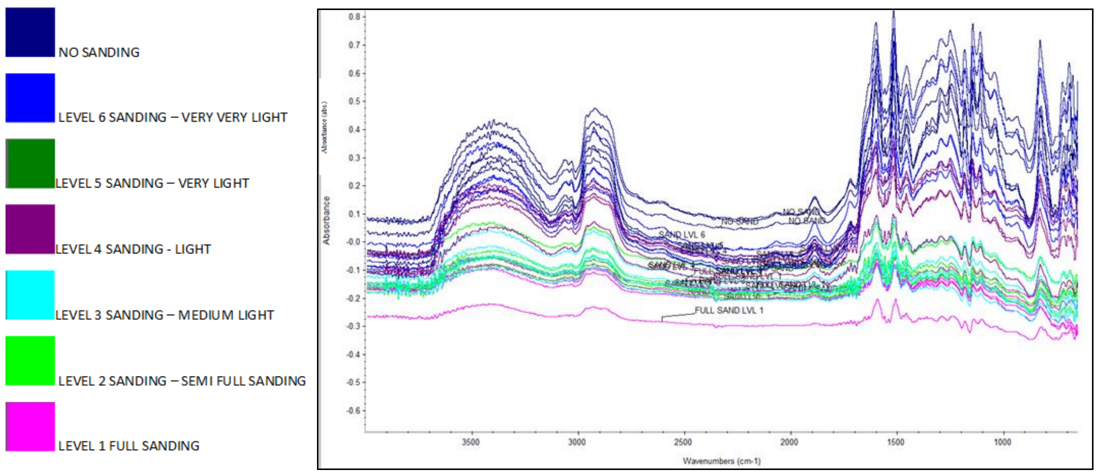

Figure 16. FTIR spectra of composite surfaces with various levels of sanding.

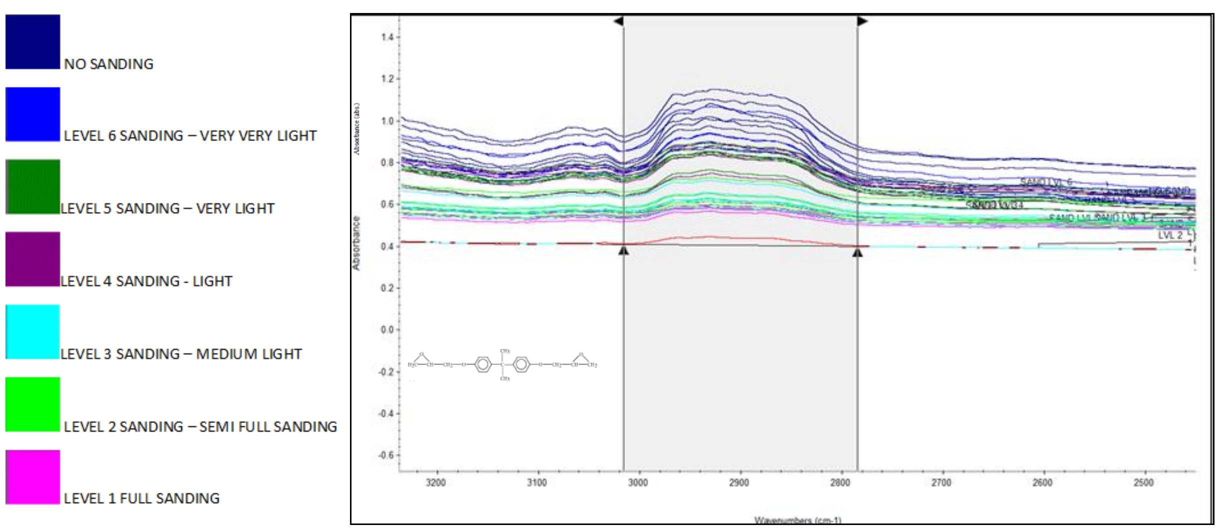

Figure 17. FTIR spectra, C-H bonding peak region, of composite with various levels of sanding.

Peak area in the C-H bonding region was plotted versus sanding level (Figure 18). Results indicated a clear decrease in peak area versus sanding level. However, there was a high standard deviation and near overlap of the error bars in the lower sanded region. There is potential for usage of FTIR for in-line process control to indicate sanded or unsanded surfaces. However, it is recommended to set a threshold limit for the amount of sanding and establish whether the peak selected has enough differentiation from the baseline values.

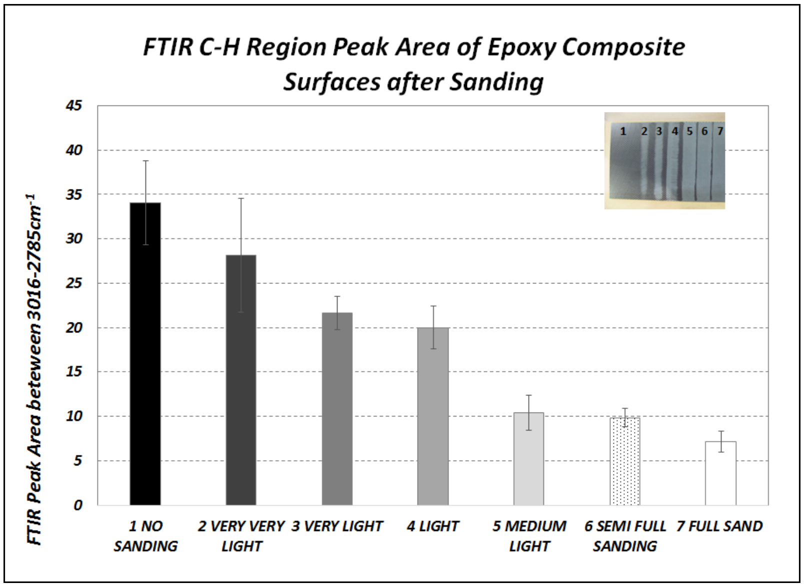

Figure 18. Detection of __level__ of sanding using FTIR C-H peak area.

3.7. OSEE

OSEE was able to successfully detect the level of surface preparation with good sensitivity (Figure 19). The signal did reach a leveling off point which should be considered when using on other substrates. Some drawbacks to OSEE was that it requires compressed argon gas to function, and there can be some sensitivity of the detector to frayed or exposed carbon fibers. However, it successfully demonstrated the ability to measure the presence and level of sanding in a rapid "go/no go" manner.

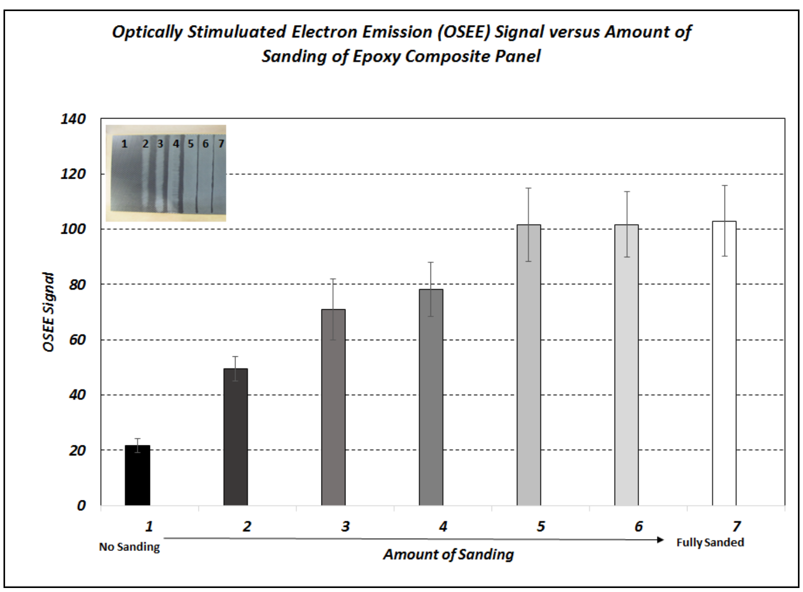

Figure 19. Detection of __level__ of sanding using OSEE.

4. CONCLUSIONS

A framework was provided that outlines a stepwise process to implement bond process control in alignment with FAA guidance. A risk analysis method was performed that demonstrated a method to identify the highest risk process parameters to target for process control. This work demonstrated that several of the analytical surface analysis tools investigated have the potential to be integrated into an in-line bond process control system. Several in-line surface analysis tools provided quantitative results that were correlated to sanding surface preparation levels: FTIR (C-H peak area), gloss (85 degree, individual maximum) and color (ΔE^* and ΔL^*, individual maximums).

Digital microscopy, WCA wettability and roughness measurement methods were not able to distinguish variation in levels of sanded surface preparations, potentially due to the surface morphology created by sanding. Other roughness measurement methods such as optical interferometry or SEM may provide more information. However, they have limited ability to be implemented as an in-line process control check tool.

Follow-on work will integrate the quantitative limits, defined from the analytical tool measurements, into the optimized bond work station. The goal is to define the processing window and control the fabrication steps to ensure a repeatable reliable bonding process.

Overall, this work has demonstrated the development of a bond process control system utilizing a selected composite substrate and sanding surface preparation. However, the work also provides a framework for implementation of bond process control on any bonded system and provides guidance to the aerospace industry on how to implement FAA recommendations and ultimately certify robust bonded structure.

5. ACKNOWLEDGEMENTS

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