Space Shuttle OV-105 Subnominal Bond Investigation

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During summer 2003, structures work along the wing/fuselage mate rivet line on NASA's Space Shuttle OV-105 (Endeavour) required thermal tile removal. The tiles were removed nondestructively, so they could be reused, by skiving through the Strain Isolator Pad (SIP) from an adjacent tile cavity. While removing the remaining SIP from the tile, technicians noted the SIP and Room Temperature Vulcanizing (RTV) was peeling adhesively from the Inner Mold Line (IML) on the tile surface. This paper presents findings that eliminate many possible causes of the subnominal adhesive bond, and suggests that the most likely cause of the adhesive bond was the use of brushes contaminated with Krylon 1201 Spray Starch or MS-143 Mold Release Agent. The research was accomplished by analyzing historical NASA documents, results of bond verification tests, peel tests, contamination tests and fabrication process anomaly tests.

Nomenclature

BHT = Butylated Hydroxytoluene
BV = Bond Verification
DMES = Dimethylethoxysilane
DR = Discrepancy Report
FTIR = Fourier Transform Infrared
GC/MS = Gas Chromatography/Mass Spectrometry
GN2 = Gaseous Nitrogen
HB = Huntington Beach
HMDS = Hexamethyldisilazane
IML = Inner Mold Line
IN = Inch
KSC = Kennedy Space Center
LB = Pound
MEK = Methyl Ethyl Ketone
N/A = Not Available
NC = Numerical Control
OCN = Order Control Number
OML = Outer Mold Line
OMRS = Operations and Maintenance Requirements and Specifications
OV = Orbiter Vehicle
PLMD = Palmdale
PRT = Problem Resolution Team
PSI = Pounds per Square Inch
PU = Production Unit
RTV = Room Temperature Vulcanizing
SIP = Strain Isolator Pad
TCA = Trichloroethane
TPS = Thermal Protection System
TPS = Test Preparation Sheet
TPSF = Thermal Protection System Facility

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I. Introduction

During summer 2003, structures work along the wing/fuselage mate rivet line on OV-105 required tile removal. In Fig. 1, the colored tiles were removed. The red tiles indicate subnominal bonds and the green tiles indicate no SIP to tile bond anomaly.

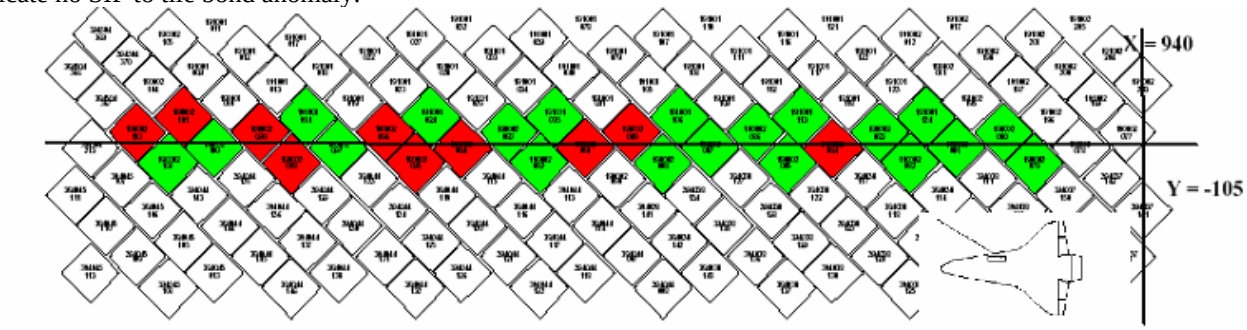

Figure 1. Tiles removed from OV-105.

The tiles were removed nondestructively, so they could be reused, by skiving through the Strain Isolator Pad (SIP) from an adjacent tile cavity. The half of the SIP that remains attached to the tile is typically removed by cutting through the SIP/Tile bond line. During that SIP removal process, technicians noted the SIP and Room Temperature Vulcanizing (RTV) was peeling adhesively from the Inner Mold Line (IML) on the surface of the tile. An adhesive peel, explained in Appendix C, is considered a subnominal bond condition, and is referred to as a subnominal SIP/IML adhesive bond.

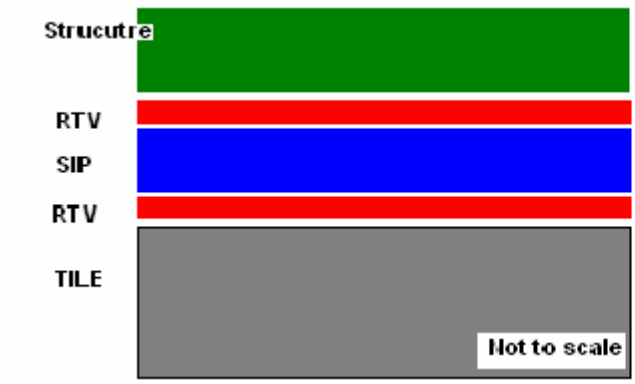

Figure 2. Cross Section of Tile Adhesive.

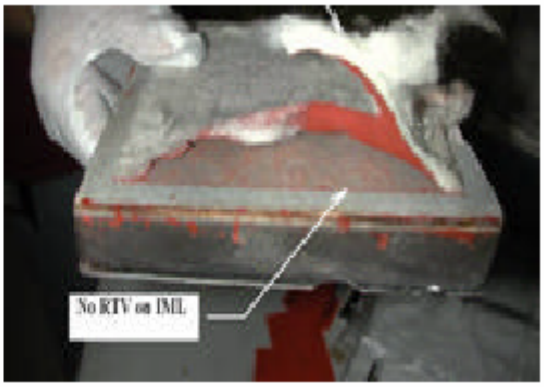

Figure 3. Subnominal Bond Failure Mode.

II. Test Overview

In an attempt to understand the possible causes of a subnominal SIP/IML adhesive bond, engineers researched historical documents for commonalities among the tiles with subnominal SIP/IML adhesive bonds. The search included, but was not limited to, a review of fabrication locations (Lockheed v Palmdale {PLMD}), fabrication dates, densification dates, technicians, methods, slurry material constituents, waterproofing dates, chemical checks, process checks, 2nd IML pre-fit dates, SIP bond dates, weather conditions, RTV lots, primer dates, and tile installation dates. Despite the widespread search and review, engineers discovered no correlation between any of those factors and the subnominal bond condition. Therefore, experiments were designed and performed to identify the root cause of the subnominal SIP/IML adhesive bonds. From an extensive fault tree analysis, engineers identified three processes which could result in a subnominal adhesive bond. These processes include slurry application to the tile IML (densification), tile waterproofing, and SIP application to the tile IML with RTV (SIP bonding). Tests were designed to analyze the effect of varying those processes on the SIP/IML bond condition. They included contamination during densification, waterproofing and SIP bonding, and changing the process variables involved in waterproofing and SIP bonding. The variables tested, chosen based on the expertise of Problem Resolution Team (PRT) members, are considered most likely to have an effect on peel strength.

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A. Initial Investigation

The original subnominal bond investigation arose from OV-105. While picture records indicate that some tiles removed from OV-103 in October 2002 have similar subnominal SIP/IML adhesive bonds as those discovered on some tiles removed from OV-105, the OV-103 anomaly was not thoroughly analyzed. No engineering investigation occurred.

Figure 4. Similar OV-103 Subnominal SIP/IML Adhesive Bond.

The investigation of OV-105's subnominal SIP/IML adhesive bond problem began with a chemical analysis to identify possible contaminants in anomalous tiles. Next, the waterproofing, were then removed based on the historical document review. The investigation also identified and investigated three processes that involved the SIP/IML interface and therefore could affect the bond strength.

Process	Description
Densification	Slurry application to the tile IML
Waterproofing	The waterproofing performed prior to SIP bond
SIP Bond	SIP application to the tile IML with RTV

Table 1. SIP/IML Interface Processes

B. Contamination Checks

Anomalous tiles from OV-105 were sent to Boeing Huntington Beach (HB) labs for contamination identification. Researchers performed a Fourier Transform Infrared (FTIR) test, allowing them to identify the presence of certain functional groups in a molecule. In a FTIR test, researchers send an energy beam through an interferometer and onto a sample. The sample absorbs and reflects certain frequencies of that beam, and a recorder captures the frequency of the energy passing through the sample in time, facilitating the derivation of the sample's chemical composition.

A Gas Chromatography/Mass Spectrometry (GC/MS) test was also performed. This test allows researchers to separate chemical mixtures based on the mass of the mo lecules and then detect and collect data showing the quantity of the various molecules collected.

C. Historical Document Review

An extensive historical document review was performed to determine if there were any process variables common to the subnominal SIP/IML adhesive bond anomaly. This search found no correlations between fabrication, processing, installation methods, locations, and techniques with the presence of the subnominal SIP/IML adhesive bonds. A complete analysis of the historical documents may be viewed in OV-105 Subnominal Bond Report[2].

D. BV Checks

Additional subsets of tiles were removed based on the historical document review and vehicle location, and Bond Verification (BV) tests were performed to assess their system strength. Ten psi BV checks were conducted using a vacuum applied to the surface of the tile, and 20 psi BV checks required bonding of the BV chuck to the tile Outer Mold Line (OML) in order to accomplish the higher loading with stress concurrence.

Figure 5. BV Check set-up.

E. Peel Tests

As there is no RTV adhesive peel requirement, this was an engineering evaluation only: a peel value greater than 4 in/lb was considered acceptable. The peels were performed using a chatillion force gauge attached with a hook to pull 1 inch strips of SIP normal to the tile IML.

Figure 6. Peel Test set-up.

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F. Staged Tests and Procedure

Two major sets of tests were created to see if process variations or contaminants introduced during densification, waterproofing, or SIP bonding would create a subnominal SIP/IML adhesive bond similar to those seen on OV-105. During the waterproofing and densification processes, major process variations and a variety of contaminants were introduced. These variations included: no waterproofing, reducing the amount of acetic acid and Silane used for various processes, and eliminating heat cleaning after waterproofing. The contaminants used were FC724 Waterproofing Compound, Trichloroethane (TCA) and Methyl Ethyl Ketone (MEK), Tri-Flo Lubricant, Krylon 1201 Spray Starch, and MS-143 Mold Release Agent. In another set of tests, the catalyst weight, RTV applied, RTV application time (catalyst drop time), RTV application time (pressure application time), and amounts of applied pressure were all varied. Engineers performed three replicate tests of each of the following factors and levels.

Factors	**Levels**
RTV catalyst quantity	1 – Double nominal amount
	2 – Nominal amount
	3 – Half of nominal amount
RTV quantity applied to tile IML	1 – Nominal amouint
	2 – Half of nominal amount
RTV application time	1 – Within potlife
	2 – After potlife expired
Pressure application time	1 – Within potlife
	2 – After potlife expired
Pressure (force) applied	1 – Contact pressure
	2 – Nominal pressure (1.5 psi)
	3 – Over pressure (3.5 psi)

Table 2. Staged Tests

Fifty-four tests were performed using TPS MISC-794-480 in the Thermal Protection System Facility (TPSF) at Kennedy Space Center (KSC). Besides the test variable, the tiles were processed normally and in accordance with the procedures. Following a full RTV cure of 7 days, the SIP on the test tiles was cut into 1-inch strips. Peel tests

were then performed in the TPSF by Boeing Materials and Processing and NASA TPS Engineering. Tiles used were retained in the SIP bond room of the TPSF for further engineering analysis.

III. Test Results

On the tiles with subnominal SIP/IML adhesive bonds from OV-105 that originally spurred this investigation, the Fourier Transform Infrared (FTIR) test revealed only silicones characteristic of RTV560/RTV566 and did not show any contaminants. The Gas Chromatography/Mass Spectrometry (GC/MS) test did not reveal any unusual data peaks, which indicates that unexpected molecules were not present. The only peak, at 13.77 minutes (retention time) had been seen on previous samples and was found in both nominal and subnominal tile samples.

This testing did not identify sources of the bond anomaly. (Note that these tiles have flown through numerous reentries. It is likely that contaminants have long since been eliminated.) The document review demonstrated that no single process deviation or material issue was the source of the subnominal SIP/IML adhesive bonds discovered on OV-105. Based on the process variables eliminated after completion of the historical document review, engineers were able to reduce possible failure causes to an unknown contaminant, a process anomaly, or degradation over time.

The BV Check and Peel Test on the initial anomalous bonds show that only 7.5% of variation in BV strength is related to peel strength. A majority of discrepant tiles had an additional BV to 10 PSI or 20 PSI prior to removal. The tensile properties are the critical design limit stress on a tile bond, so it is favorable that all subnominal peel strength bonds still passed a BV check. While a typical peel strength for a nominal tile bond is greater than 4 lb/in , anomalous tiles revealed a peel strength as low as 0.5 lb/in.

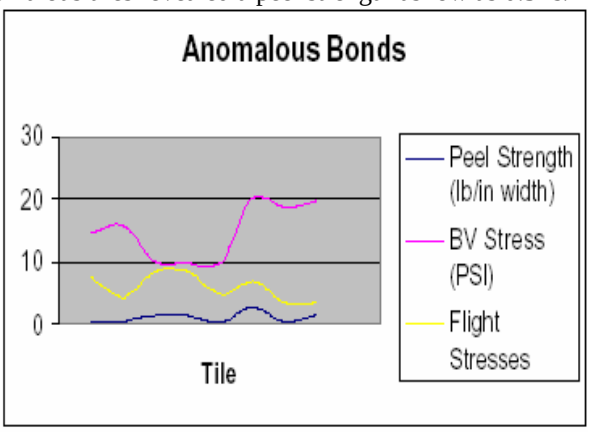


Figure 7. Anomalous Bond Tiles.

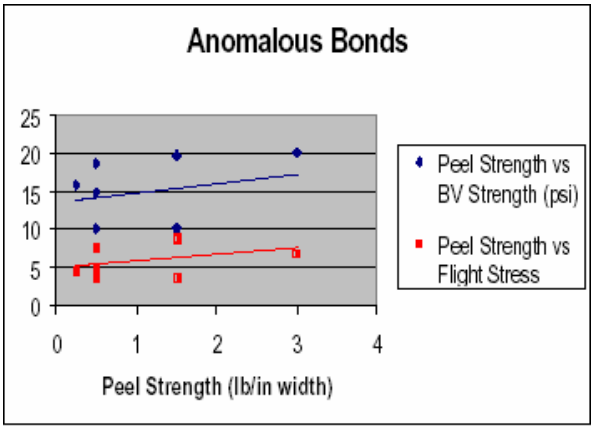
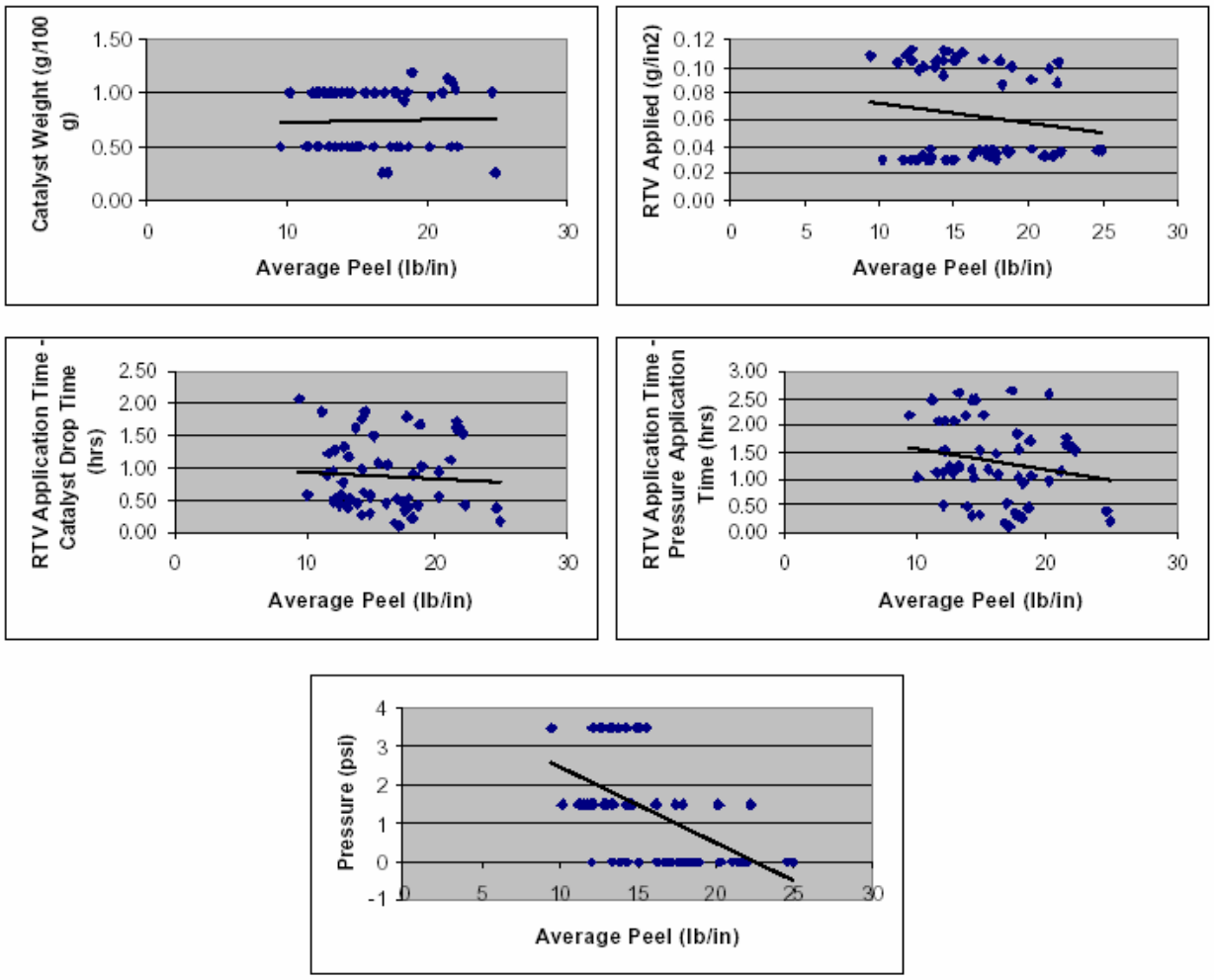


Figure 8. Anomalous Bond peel strength.

A. Staged Test Results

The catalyst weight had no effect on the peel strength, but decreasing amounts of RTV applied, RTV application time, and application pressure decreased the peel strength on a batch of tiles processed per MISC-794-480. The effect of decreased pressure produces the most extreme results.



Figures 9-13. Staged test results for fabricated orbiter tiles.

The at which the SIP/IML bond is exposed to a contaminant is not a factor in its peel strength, as long as its exposure is prior to the SIP and IML actually becoming bonded. While a significant deviation from the written waterproofing process, such as not adding silane, would cause a subnominal bond, data indicates that subnominal bonds induced by process variations were not nearly as extreme as those discovered in OV-105. Additionally, the document review revealed that it is very unlikely that such an extreme waterproofing process variation could have occurred. The contaminants that caused the most extreme reduction in peel strength were Krylon 1201 pray Starch and MS-143 Mold Release Agent. Additionally, as the amount of contaminants added increased, the peel strength decreased.

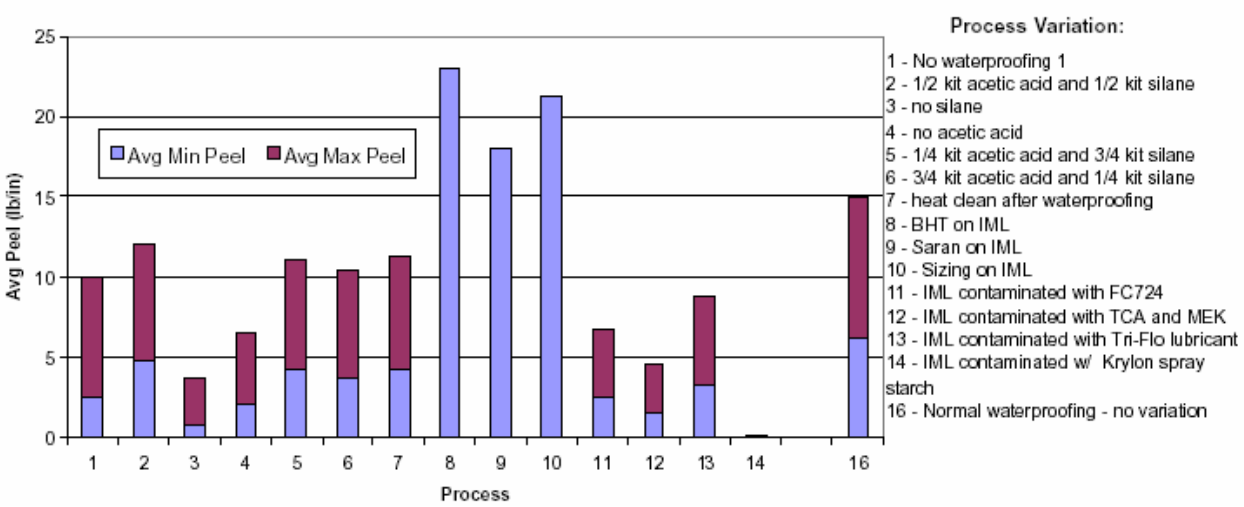


Figure 14. Peel for MISC-794-479

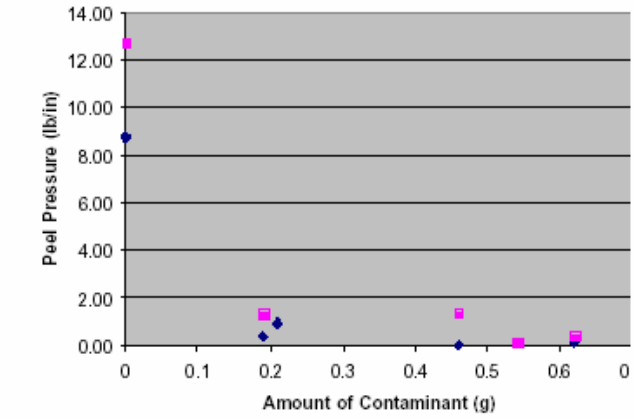


Figure 15.IML Contaminated with Krylon Spray Starch

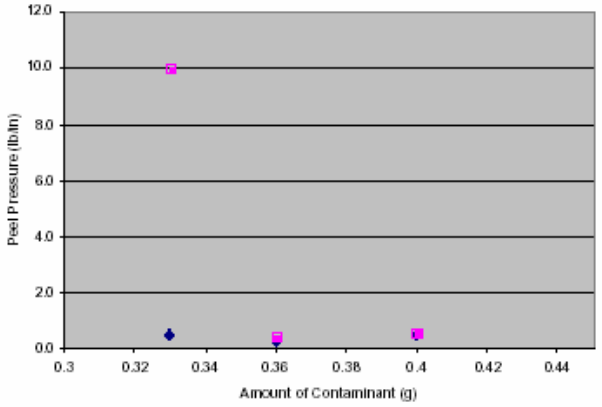


Figure 16. IML Contaminated with MS-143 Mold Release Agent

IV. Summary of Results

After the historical document review, the possible causes for the subnominal SIP/IML adhesive bond were limited to: oven pump malfunction; densification material anomalies; factory waterproofing material anomalies; SIP bond process environmental conditions; SIP bond process workmanship; densificatio n workmanship; densification process deficiency; factory waterproofing workmanship; SIP bond process contamination; vehicle location; and age issues.

A. Oven Pump Malfunction

An option that can be eliminated as a possible cause of a subnominal SIP/IML adhesive bond is that the oven pump malfunctioned, causing inadequate waterproofing. While poor waterproofing does reduce the peel strength, applying no waterproofing at all does not produce peel strengths that even approach the low value of the subnominal peels observed on OV-105.

Figure 17. Peel without waterproofing (avg peel = 13.5 lb/in).

B. Densification Material Anomalies

The possibility of a densification material anomaly remains open and test results are not available to show that the material was composed properly and not contaminated.

C. Factory Waterproofing Material Anomalies

The silane used in the factory waterproofing process could have been impure. This remains an option as silane is an integral part of producing a nominal SIP/IML bond as demonstrated in the test peels for MISC-794-479. However, the peel strength values are still not nearly as low as those observed in the subnominal SIP/IML adhesive bonds on OV-105.

Figure 18. Peel without silane (avg peel = 6 lb/in).

Figure 19. Normal peel (avg peel = 22lb/in).

D. SIP Bond Process Environmental Conditions

The environmental data available was minimal at best. Weather data was obtained from Edwards AFB, more than 60 miles from the processing facility at Palmdale. The time span during which the SIP/IML bond could have been affected had the conditions shown in Table 3.

	Minimum	**Maximum**
RH Level	14.3	100
Temp	-11.0°C	39.9°C
Precipitation	0.0 in	0.65 in

Table 3. Environmental conditions.

No data was available that could compare the actual SIP/IML bond fabrication date to the ambient weather conditions on that date. The densification procedure states that the environment must be "such that the work area will be maintained generally clean, with housekeeping provisions to minimize dust, dirt, lint, and other airborne contaminants" (MPP 609M303M01 p5)[1].

E. SIP Bond Process Workmanship

SIP bond process workmanship is another issue unresolved by the document search. However, a subset of that workmanship, application of the wrong catalyst quantity, can be eliminated as a possible cause because of the tests revealing that catalyst amount had very little effect on bond peel strength.

Figure 20. Peel with .25g (minimal) Catalyst

Figure 21. Normal Peel

Yet, it remains a possibility that the RTV could have been incorrectly applied, though the effect on the peel strength is not as great as on the subnominal SIP/IML adhesive bonds identified on OV-105.

Figure 22. 3.61g RTV Applied

Figure 23. 0.78g RTV Applied

F. Densification Workmanship/Process Deficiency

Densification workmanship and densification process deficiency as possible causes can be attributed to the same factor: contaminated brushes. The brush cleaning instructions do not dictate how frequently the alcohol bath should be changed when single brushes are being cleaned. This facilitates contamination. Should the brushes became contaminated with Krylon 1201 Spray Starch or MS-143 Mold Release Agent, the peel strength could reduce to subnominal SIP/IML adhesive bond levels.

G. Factory Waterproofing Workmanship/SIP Bond Process Contamination

Waterproofing workmanship contamination and SIP bond process contamination could have the same results as brush contamination. Krylon 1201 Spray Starch and MS-143 Mold Release Agent are two contaminants that are common in tile processing facilities and therefore could have tainted the purity of the tile IML. Both of these contaminants reduced peel strength to levels similar to those observed when the adhesive failure anomaly was seen on OV-105. Based on information available, such contamination is the most likely cause of the adhesive bond failure.

Figure 24. Peel with Krylon 1201 Spray Starch.

Figure 25. Peel with Mold Release Agent.

H. Vehicle Location

All failure bonds were Palmdale bonds. Whether it was a contaminant, weather conditions, processing anomalies, or other unexplained factors at that location that led to these failures remains unknown. However, the volume of tiles processed at Palmdale is exponential as compared to those processed at KSC; therefore, the small number of subnominal SIP/IML adhesive bonds emerging from Palmdale remains statistically insignificant.

I. Age Issues

Analysis and document review has neither eliminated nor advanced the possibility that the SIP bond degraded over time.

V. Recommendations/Conclusions

The following are suggestions to help eliminate future subnominal SIP/IML adhesive bonds and better understand their cause.

The labs at KSC and HB did not fully coordinate subnominal bond research, and contaminant peel tests were conducted under different conditions. No repeatable procedure was available for the data acquired from HB. In order to accurately gauge the affect of Saran, Sizing, and BHT contamination on the SIP/IML bond, those tests should be recreated under standardized, controllable conditions and in a manner such that they can be properly compared to other contamination investigations. Additionally, future testing at multiple facilities should be coordinated by all parties involved to avoid inefficacious results.

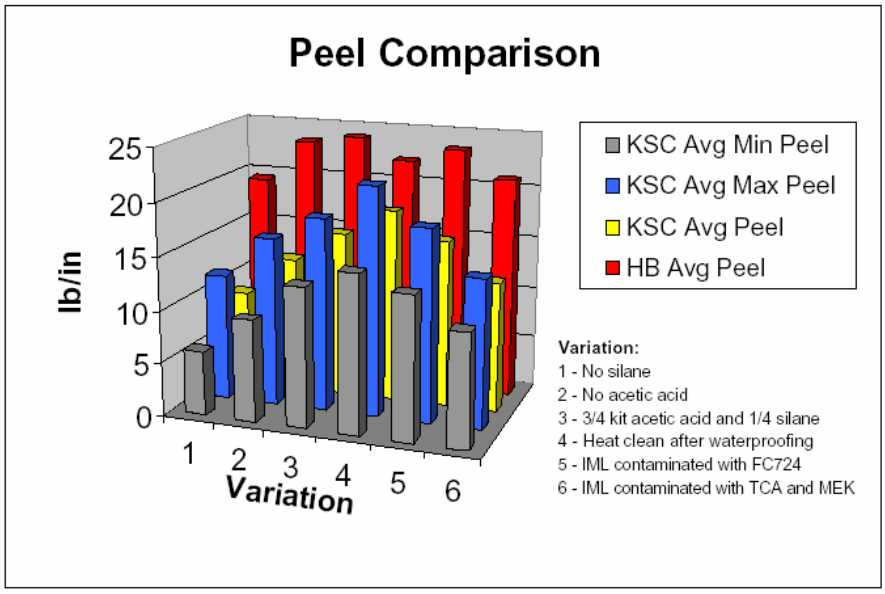


Figure 26. Comparison of testing at different facilities.

If Krylon 1201 Spray Starch or MS-143 Mold Release Agent were the cause of the subnominal SIP/IML adhesive bond, it is most likely that they were introduced to the system by a contaminated brush. The current densification procedure calls for brush cleaning before slurry application. The procedure should be modified to include an additional brush cleaning after slurry application to prevent used brushes from becoming further contaminated by lying around, covered with slurry, for an indefinite time between applications. Additionally, guidelines should be added to outline how often the cleaning alcohol bath should be replaced in all situations. A log should be created to help technicians track when the alcohol bath is changed.

Because of the affect they have on bond strength, Krylon 1201 Spray Starch and MS-143 Mold Release Agent should not be allowed in the vicinity of tile prior to the SIP and IML becoming bonded.

Check all removed tiles for indications of a subnominal SIP/IML adhesive bond anomaly to monitor the problem over time. If the problem begins to emerge at an increased rate, a more extensive study of age degradation will be necessary. Continue research on this issue, to include monitoring OV-103's possible subnominal SIP/IML adhesive bonds.

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[1] "Procedure for Densification: MPP 609M303M01," *NASA*, p 5.
[2] Gunn-Golkin, A., "OV-105 Subnominal Bond Report," *NASA KSC PH-H!* (to be published).