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EVALUATION OF SPACE SHUTTLE TILE SUBNOMINAL BONDS

by

COOPER GRIFFIN SNAPP B.S. University of Kansas, 2000

A thesis submitted in partial fulfillment of the requirements for the degree of Master of Science in the Department of Mechanical, Materials, and Aerospace Engineering in the College of Engineering and Computer Science at the University of Central Florida Orlando, Florida

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ABSTRACT

This study researched the history of Space Shuttle Reusable Surface Insulation which was designed and developed for use on the United States Orbiter fleet to protect from the high heating experienced during reentry through Earth's atmosphere. Specifically the tile system which is attached to the structure by the means of an RTV adhesive has experienced situations where the bonds are identified as subnominal. The history of these subnominal conditions is presented along with a recent identification of a subnominal bond between the Strain Isolation Pad and the tile substrate itself. Tests were run to identify the cause of these subnominal conditions and also to show how these conditions were proved to be acceptable for flight.

The study also goes into cases that could be used to identify subnominal conditions on tile as a non-destructive test prior to flight. Several options of non-destructive testing were identified and recommendations are given for future research into this topic.

A recent topic is also discussed in the instance where gap fillers were identified during the STS-114 mission that did not properly adhere to the substrate. The gap fillers were found protruding past the Outer Mold Line of the vehicle which required an unprecedented spacewalk to remove them to allow for a safe reentry through the atmosphere.

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LIST OF ACRONYMS/ABBREVIATIONS

AETB	Alumina Enhanced Thermal Barrier
AFB	Air Force Base
AFRSI	Advanced Flexible Reusable Surface Insulation
ВНТ	Butylated Hydroxytoluene
BRI	Boeing Rigidized Insulation
BV	Bond Verification
DMES	Dimethylethoxysilane
ET	External Tank
FI	Flexible Insulation
FRCI	Fibrous Refractory Composite Insulation
FRCS	Forward Reaction Control System
FRSI	Flexible Reusable Surface Insulation
FTIR	Fourier Transform Infrared
GC	Gas Chromatography
HB	Huntington Beach
HRSI	High-Temperature Reusable Surface Insulation
IML	Inner Mold Line
IP	In Plane
KSC	Kennedy Space Center
LESS	Leading Edge Structural Subsystem
LI	Lockheed International

LRSI	Low-Temperature Reusable Surface Insulation
MD	Master Dimension
MEK	Methyl Ethyl Ketone
MPP	Material Processing Procedure
MR	Material Review
MS	Mass Spectormetry
NASA	National Aeronautics and Space Administration
NC	Numerically Controlled
OCN	Order Control Number
OML	Outer Mold Line
OMS	Orbital Maneuvering System
OPF	Orbiter Processing Facility
OV	Orbiter Vehicle
Pcf	Pounds per Cubic Foot
PLMD	Palmdale
Psi	Pounds per Square Inch
PTFE	Polytetrafluoroethylene
RCC	Reinforced Carbon-Carbon
RCG	Reaction Cured Glass
RCS	Reaction Control System
RSI	Reusable Surface Insulation
RTV	Room Temperature Vulcanizing
SIP	Strain Isolator Pad

SPC	Spaceflight Processing Contract
SRB	Solid Rocket Booster
SSME	Space Shuttle Main Engine
TCA	Trichloroethane
TEOS	Tetraethylorthosilicate
TPS	Thermal Protection System
TPSF	Thermal Protection System Fabrication
TTT	Thru the Thickness
TUFI	Toughened Unifibrous Insulation
USA	United Space Alliance
VAB	Vehicle Assembly Building
VHT	Very High Temperature
WLE	Wing Leading Edge

1.0 SPACE SHUTTLE THERMAL PROTECTION SYSTEM OVERVIEW

The Thermal Protection System (TPS) of the Space Shuttle Orbiter is unique when compared to other atmospheric reentry vehicles in that it, along with other Orbiter subsystems, is reusable. Of these reusable systems, TPS is unique because the existing design concepts from the aerospace industry could not be utilized in its original development. During a typical reentry heating cycle, the orbiter is subjected to temperatures in excess of 2,300°F as shown in *Figure 1*. The mostly ceramic-based TPS protects the orbiter aluminum and payload bay door graphite epoxy structure and its penetrations from reaching temperatures over 350°F, and the Orbiter Maneuvering System (OMS) pod graphite epoxy structure from exceeding 250°F. The Shuttle TPS is more than tiles and blankets, as would be the definition from the casual observer. TPS is the integration of all of the materials, development, design concepts, fabrication techniques, installation processes, and refurbishment procedures used to protect a vehicle from the severe heating environment of atmospheric reentry.

The principle design functions of the TPS are to perform as a radiator (to emit heat), a reflector (to prevent on-orbit heating), and as an insulator (to protect the structure from the residual heat flux). The TPS is primarily white on the upper surface and black on the lower surface to control on-orbit heating from solar radiation and to maximize heat rejection during reentry. By rotating the orbiter so that the more reflective (and less absorbent) white upper surface is towards the sun, the solar heating is minimized. Conversely, directing the black lower surface towards the sun would maximize the solar heating. The high emissivity black region must be on the lower surface to maximize the heat rejection (in the form of thermal radiation) from the TPS during reentry where this region experiences the highest heat load.

In addition to protecting to structure from heat loads up to 66,000 Btu/ft², the outer mold line (OML) of the TPS serves as the aerodynamic shape of the vehicle. This shape is maintained by tight control of the step and gap between installed TPS components. Excessive steps and/or gaps between parts can result in early transition of the laminar to turbulent boundary layer which would result in higher heat loads. Minor steps and/or gaps can result in local overheating which could slump (i.e., melt and deform) tiles or permit subsurface plasma flow, which, in turn, could degrade the TPS bondline or underlying structure.



Figure 1 - Maximum Recorded Surface Temperatures - STS-1 through STS-5

The Shuttle TPS must also protect the structure from localized heating from plumes of the Space Shuttle main engines (SSME), solid rocket boosters (SRB), OMS engines, and reaction control system (RCS) thrusters. In addition to the thermal demands, the TPS also withstands the launch acoustics (up to 166 decibels), structural deflections from aerodynamic loads, on-orbit cold soak temperatures (down to -250°F), environmental exposure at the ocean-side launch pads, and potential damages associated with ground processing.

The primary materials which make up the TPS are as follows:

- Reusable Surface Insulation (RSI) Tiles
- Flexible Insulation (FI) Blankets, originally developed as Advanced Flexible Reusable Surface Insulation (AFRSI)
- Felt Reusable Surface Insulation (FRSI)
- Reinforced Carbon-Carbon (RCC)
- Gap Fillers
- Thermal Barriers
- Thermal Seals
- Window Thermal Panes

The approximate locations of these materials are given in *Figure 2* and the specific discussions of each of the materials are provided in the following sections.



Figure 2 - Space Shuttle Orbiter TPS Configuration

1.1 Reusable Surface Insulation (RSI) Tiles

On average there are 24,300 RSI tiles installed on each operational vehicle. It should be noted that there were slightly more tiles on OV-102 (Columbia) due to its original TPS configuration predating flexible blanket technology. RSI tiles are made from one of five substrate materials (LI-900, FRCI-12 insulation, and LI-2200, AETB-8 and BRI-18) and are coated with a white or black glass coating. White-coated RSI tiles are referred to as Lowtemperature Reusable Surface Insulation (LRSI) and black-coated tiles are known as Hightemperature Reusable Surface Insulation (HRSI). LRSI is used in areas where the peak temperatures do not exceed 1,200F, and HRSI is used in regions less than 2,300°F.

The tile substrate material and coating selection are dependent on the mechanical and thermal requirements of the particular location. For example, tiles located on the upper surface of the forward fuselage (some of which are 0.75-inch thick LI-900 LRSI) experience much lower temperatures and require less strength than tiles on the nose landing gear door (which are 2 to 3inch thick FRCI-12 and LI-2200 HRSI). The thickness of the tiles varies with heat loads and OML contour requirements from less than 1 inch to over 3 inches. The substrate material is machined to the desired shape (usually 6 inch by 6 inch by necessary thickness) prior to coating. The tiles are mostly located on the lower surface on the vehicle, as they have a greater resistance to high heat loads and provide a smoother, more aerodynamic surface than flexible blankets.

Figure 3 depicts a typical RSI tile installation. All of the tiles are bonded to the structure using strain isolator pads (SIP) and room temperature vulcanizing (RTV) silicone adhesives. The IML of the RSI tile is densified prior to SIP bond to uniformly distribute stress concentration

loads at the tile-to-SIP interface. The structure beneath tile-to-tile gaps is protected by filler bar. Gap fillers are used in areas of high differential pressures, extreme aero-acoustic excitations, and to passivate over-tolerance step and gap conditions.



Figure 3 - Typical RSI Tile Installation

RSI tiles require rewaterproofing prior to each mission because the waterproofing compound degrades at temperatures exceeding 1,050°F. The rewaterproofing is accomplished by the injection of at least 2ml of dimethylethoxysilane (DMES) waterproofing compound into each tile. The DMES renders the tile substrate hygrophobic by reactions between the Si-OH groups in the silica and the ethoxy group in the DMES with negligible weight gain. Failure to rewaterproof RSI tiles could result in increased weight (from absorbed water) or tile damage. The damage would be caused by the absorbed water freezing and subsequently contracting on orbit at cold soak temperatures below -70°F, thereby inducing a fracture at the 1,050°F isotherm. During reentry, the absorbed water would convert to steam and complete the failure of the tile by loss of the dewaterproofed region previously fractured (Refer to **Error! Reference source not found.**). In addition to thermal exposure, the silylated (i.e., waterproofed) surfaces that are not protected by the original tile coating (i.e., damaged or previously repaired areas) could degrade from exposure to atomic oxygen attack on orbit.



Figure 4 - Tiles with Coating Damage due to Water in Tiles during STS-2

1.1.1 RSI Tile Substrate Materials

There are five RSI tile substrate materials currently used on the orbiter, 9 and 22 pcf Lockheed Insulation (LI-900 and LI-2200), 12 pcf Fibrous Refractory Composite Insulation (FRCI-12), 8 pcf Alumina Enhanced Thermal Barrier (AETB-8), and 18 pcf Boeing Rigidized Insulation (BRI-18). The LI-900 and LI-2200 materials are comprised of high-purity amorphous silica fiber (LI-2200 adds a small amount of silicon carbide powder) made rigid by ceramic bonding. The FRCI-12 material is similar to the LI-2200 except aluminoborosilicate fiber is added to the silica fiber and silicon carbide powder. The FRCI-12 material is made rigid by boron fusion at the fiber junctions. The AETB-8 and BRI-18 are similar composition but are coated with an impact resistant Toughened Unifibrous Insulation (TUFI). This high impact coating helps protect the tile from being damaged from an impact. The fabrication of all five materials is similar, and is shown in *Figure 5*.



Figure 5 - Fabrication Schematic for RSI Tile Substrate Materials

The fabrication of LI-900 is accomplished in six basic steps. The 99.7% pure silica fiber is dispersion washed in deionized water. The fiber is mixed with Ludox ammonia stabilized colloidal silica solution in a V-blender for a specific duration to obtain the proper length of fibers. The fiber slurry is removed from the V-blender and is poured into a casting tower where excess liquid is removed. The mixture is then pressed in the casting tower to a specific height that will yield the desired dry density. The block is removed from the casting tower and is placed in a low-temperature oven to dry. The dried block is then sintered at a specific high temperature and duration to activate the ceramic bonding and yield the desired final density.

The fabrication of LI-2200 is identical to the procedure for LI-900 except 1200 grit silicon carbide powder is added (3% by weight) to the silica fiber (97% by weight) prior to mixing in the V-blender. The silicon carbide is used to improve the emissivity of the LI-2200 material.

The fabrication of FRCI-12 is similar to LI-2200, with the exception of an additional calcining step for high-boria content aluminoborosilicate fiber (62% alumina/14% boria/24% silica) at 2,200°F for 90 minutes. The calcined aluminoborosilicate fiber is added (21.5% by weight) to the silica fiber (76.5% by weight) and silicon carbide powder (2% by weight) prior to mixing in the V-blender. During the sintering of the material, the boron content in the aluminoborosilicate fuses the fibers together at the junctions resulting in a more rigid structure than the LI-900 and LI-2200 ceramic bonding.

The physical properties and use temperatures of the various substrate materials is given in *Table 1*.

Property	LI-900	LI-200	FRCI-12
Density (pcf)	8.75 +/- 0.75	22.0 +/- 2.0	12.5 +/- 1.0
IP Tensile Strength (psi)	68	120	141
TTT Tensile Strength (psi)	24	50	52
Thermal Conductivity at 0.001atm (Btu*in/ft ² *hr* ^o F)	0.021	0.03	0.027
Use Temperature (°F)	2,300	2,300	2,300

Table 1 - Properties of RSI Substrate Material

1.1.2 RSI Tile Modeling and Machining

There are two distinctly different types of tile machining, tracing a physical model of the cavity on a stylus machine to produce a flight tile or using a numerically controlled (NC) milling machine to create a tile based on a three-dimensional computer model. The use of either method is dependent on the modeling technique employed.

There are four modeling techniques used, a cavity tracer pattern splash per ML0601-9024 process 102, manual computer modeling from master dimension data per process 317, automatic computer modeling on the floor (i.e., by technicians working on the orbiter in the OPF) using tile cavity digitization, and automatic computer modeling on the floor using the Optigo tile digital scanning device.

Following the necessary signatures to authorize the work, the tile is ordered. If applicable, the floor-level cavity modeling is performed by the United Space Alliance (USA) technicians, specifically cavity splashes, cavity digitizing, and Optigo picture frame fabrication. A tile traveler (i.e., form used to obtain a replacement tile) is issued and, with any additional items (tracer patterns, computer data, or mylars of the cavity), is forwarded to the Thermal Protection System Facility (TPSF) for further processing.

Splashes involve the fabrication of a tile from a tracer pattern using a physical model of the cavity. A tracer pattern is made from a polyisocyanate and polyurethane foam casting of the open cavity. The OML is faired to be flush with the adjacent RSI and, as a result, the technician performing the splash approximates some of the design features, such as the contour of the OML. As a result, the Master Dimension (MD) configuration of the orbiter is oftentimes not

maintained. Drawing defined features, such as delta lips, are not modeled on the tracer pattern. Instead, the sidewall lip is noted on the pattern and the lip is machined to theoretical dimensions following the machining from the tracer pattern. Splashing a tile cavity is a time consuming process, which can take up to a full shift to produce a tracer pattern. Despite the time consumption, splashes can be performed on all cavities and it is an efficient method for modeling sidewall jogs and other non- design features.

Splashes are convenient in that they provide a real-time determination of fit to adjacent tile and, as a result, minimal step and gap rework is required for the bonded replacement tile. The machining of the tile from a tracer pattern is a less accurate process than NC machining as the tracer stylus often chatters on the tracer pattern resulting in poor dimensional stability. The materials that comprise the tracer pattern require the technicians to wear protective equipment. In addition, the tracer patterns are extremely moisture sensitive and can degrade while in storage in the Vehicle Assembly Building (VAB). Therefore, the storage of these patterns results in costly inventory which, oftentimes, returns an unusable product.

The NC machining is a more precise method using current technology such as automation and, in some cases, optical modeling. The cavity models can be created in a variety of ways, but most methods utilize theoretical MD data. Therefore, for most NC related processes, the MD configuration of the orbiter is maintained. NC models can be made of any cavity. The cavity models are saved electronically, whereby they are easy to recall, modify, and used to recreate tiles. One of the disadvantages of NC machining is that the initial programming can be time consuming, especially if sidewall jogs or non-design features are required. This time is easily offset for a recurring replacement tile, such as a landing gear door corner tile or a tile adjacent to a Reaction Control System (RCS) thruster. Sometimes, the theoretical tile Inner

Mold Line (IML) does not correspond to the vehicle structural configuration. In these cases the NC tile does not fit without corrective rework. Another disadvantage of NC machining is the cost of the associated hardware, but this cost is offset by the time saved in modeling and the high quality of the finished product. NC models are saved as electronic data which require storage on magnetic media. Provided the media is kept in an office environment, the models are highly reliable.

In accordance with the standard process for the installation of replacement RSI tiles (ML0601-9024 process 301), the coated, undensified tile is sent to the USA technicians for prefit into the cavity and is evaluated for conformance with the installation step and gap criteria outlined in the ML0601-0001 specification and engineering drawing. If the tile is acceptable per this evaluation, the tile fabrication process continues with IML densification, waterproofing, and SIP bonding. If the evaluation indicates the tile is not within the installation requirements, the tile is sent to engineering for further disposition. Refer to *Figure 6* for a graphical representation of the modeling, machining, and evaluation processes.



Figure 6 - RSI Tile Modeling and Machining Flow Diagram

1.1.3 RSI Tile Coating, Factory Waterproofing and IML Densification

There are two types of RSI tile coating materials, a white (for LRSI tiles) and a black (for HRSI tiles) glass coating. Either of the two coatings can be applied to the five substrate materials, and is governed by the engineering drawings. The white coating is completed by a seal coat, top coat, and firing process. A 10%-Ludox ammonia stabilized colloidal silica/deionized water seal coat solution is sprayed on the tile Outer Mold Line (OML) and sidewalls, leaving the terminator vent zone (an area approximately 0.2 inch above the tile IML) uncovered. The seal coat is dried and the tile is heat cleaned at 1,100°F to 1,450°F for 10 minutes. One coat of the water-based borosilicate glass slurry/acrylate thickening agent top coat is sprayed on the tile. The first coat is air dried and a second coat is sprayed. While the second coat is wet, the tile is oven dried at 1,150°F for 30 minutes. The tile is sintered at 2,100°F for 70 minutes. The fired coating weight is 0.07 to 0.17 lb/ft2 and the coating thickness is 0.007 to 0.011 inch.

The black reaction cured glass (RCG) coating is accomplished by a top coat and firing process. The RCG slurry contains powdered borosilicate glass frit, tetraboron silicide powder, and a methylcellulose suspension agent in a denatured alcohol carrier. The tile is heat cleaned at 1,100°F to 1,450°F. The tile is wetted with denatured alcohol and sprayed with 9 to 13 coats of the RCG slurry. The coating is air dried for 3 hours and the tile is sintered at 2,215°F for 95 minutes. The fired coating weight is 0.09 to 0.17 lb/ft2 and the coating thickness is 0.009 to 0.015 inch.

Each of the tiles is identified using a black or white very high temperature (VHT) paint in the opposing color to the tile coating. The identification includes the part number from the engineering drawing, the order control number (OCN) to provide traceability, and any other necessary markings (e.g., instrumentation markings, MR designations, etc.).

All of the RSI tiles require factory waterproofing. The original waterproofing is accomplished by the vapor deposition of methyltrimethoxysilane. The tile is placed in a vacuum deposition oven heated to 350°F and is held at a minimum of 27 in. Hg. Heated acetic acid is first injected into the vacuum chamber followed by heated methyltrimethoxysilane. The silane renders the tile substrate hygrophobic by reactions between the Si-OH groups in the silica and the ethoxy group in the silane with negligible weight gain. A minimum of 0.2% silane weight pickup is required to accept the tiles as being waterproofed.

The IML surface of every RSI tile is densified to evenly distribute stress concentrations at the SIP-to-tile interface. The densifying material consists of a mixture of dispersed ground highpurity silica into a Ludox ammonia stabilized colloidal silica solution and tetraboron silicide. The waterproofed IML area is wetted with isopropyl alcohol and the material is applied to a specific weight pickup per unit area. The tile is air dried for 24 hours and then the tile is heated at 400°F for 2 hours to remove any residual acetic acid from the waterproofing process.

1.1.4 RSI Tile Bondline / Subsurface Components

The tiles are bonded to the structure via a strain isolator pad (SIP). The majority of SIP is a non-heat-treated Nomex polyaramid felt pad. The discussion of the Nomex material is given in Section 1.0. The SIP is available in three thicknesses, 0.090, 0.115, and 0.160 inch. The 0.090inch SIP is used for high-modulus bonding applications, such as adjacent to thermal barrier installations (where the tile encounters side loading in addition to flight loads). The 0.160-inch SIP is commonly used in acreage applications. The SIP is bonded to the tile IML following the densification and vapor deposition waterproofing operations. The SIP is bonded to the IML with a RTV silicone adhesive under vacuum pressure. In most situations, the SIP periphery is located one-half inch within the periphery of the tile IML to allow access for the filler bar installation on the structure. The SIP-bonded tile is routed for cavity installation.

The filler bar is bonded to the structure beneath the tile gaps. Filler bar is also used in a similar fashion for FI blanket installations. The heat-treated Nomex felt strips (usually 0.75 inch wide) are bonded with RTV silicone adhesive under pressure in a lattice pattern prior to RSI tile installation. The filler bar provides thermal insulation to the structure from hot plasma flow into the tile gap. The filler bar also provides a seal between the structure and tile IML, protecting the tile bondline. The filler bar can withstand 800°F topside exposure.

In certain regions of the orbiter, pre-cast RTV silicone heat sinks are installed beneath the bondlines of RSI tiles, FI blankets, or FRSI. The heat sinks are used to uniformly distribute backface heat loads to reduce thermal gradients within the orbiter structure. To compensate for mismatches at structural interfaces or around fasteners, a RTV silicone adhesive (screed) is used to fill voids and provide a smooth surface for RSI bonding.

1.1.5 RSI Tile Removal and Installation

RSI tiles are occasionally removed and replaced as a part of routine TPS maintenance. The reason for the replacement could be in support of a new tile installation on a new vehicle, or more realistically, in support of a TPS reconfiguration modification. Tiles are also removed and replaced due to severe damage or material degradation of the part. A flow diagram of the replacement process is shown in *Figure 7*.



Figure 7 - RSI Tile Replacement Flow Diagram

The RSI tile is removed either destructively or non-destructively per ML0601-9024 process 300. Usually, a knife is used to cut through the SIP to remove the tile non-destructively

for future use. If the non-destructive method can not be used, the tile is carefully broken into pieces and removed from the bonded SIP. The remains of the SIP and the residual RTV are skived off with a non-metallic scraper and the tile IML is solvent cleaned.

The tile installation is performed per ML0601-9024 process 301. The tile cavity is modeled and a flight tile is machined (refer to Section 1.1.2). The tile is coated with either the white glass or black RCG coating and is identified (refer to Section 1.1.3). The tile is routed to the USA technicians for first prefit in the tile cavity. The prefit is used to fit-check the tile and evaluates any step or gap discrepancies that may exist (refer to *Figure 3*). The IML mismatch to the structure is also verified to be within close tolerances prior to continuing the tile processing.

Following the acceptance of the first prefit, the tile and its cavity can be processed in parallel. The tile IML is densified and the tile is waterproofed (refer to Section 1.1.3). The tile is prefit a second time to verify conformance prior to SIP bond. During the same time as the tile processing, the cavity is prepared. Substrate voids, if any, are filled with a RTV silicone adhesive. The filler bar is installed or reworked as required per ML0601-9024 process 215. The substrate is cleaned per the applicable process (ML0601-9024 process 200-207) and is primed with a silicone primer per process 208.

The SIP is bonded under 1 to 3 psi pressure to the tile IML for 0.160-inch SIP and 2 to 3 psi for 0.090/0.115-inch SIP (refer to Section 1.1.4). Two customized bonding tools are fabricated, the tile pressure pad and the bond verification (BV) chuck. The pressure pad is a latex foam pad which is calibrated with the required density and thickness for the installation and bonded to a rigid block that matches the OML contour of the tile. The block interfaces between the reaction tooling and tile during bond pressure application. The geometry of the pressure pad directs the pressure uniformly about the tile centroid. The BV chuck is a rigid block that

matches the tile OML contour and has a gasket around the OML periphery. The chuck is used to draw a vacuum across the OML surface for tensile testing following adhesive cure.

The tile is prefit a final time and an "OK to install" is obtained when all previous processing has been completed. The tile is bonded with RTV silicone adhesive under 1 to 3 psi pressure as directed through the pressure pad and reaction tooling. Proper pressure is verified by measuring the compressed foam thickness at each corner. Following the cure of the adhesive, the pressure is removed and the bond is tested by a bond verification tensile test per ML0601-9024 process 315. The BV chuck is pressed to the tile OML and a vacuum is drawn through the chuck. The chuck is attached to a threaded shaft or cable assembly to the tensile test unit. The tile is loaded in tension (10 psi of SIP-bonded area for LI-2200 and FRCI-12 and 4 to 6 psi for LI-900) until the specified load is reached. This loading is reduced or eliminated as directed by the engineering drawing for structurally limited areas (e.g., vertical stabilizer, OMS pod). Following bond verification, the step and gap are measured and are verified to be within the ML0601-0001 operational criteria. Following acceptance, the tile installation is complete. Gap filler installation, if required, is performed at this time (refer to Section
1.5 Gap Fillers).

1.2 Flexible Insulation (FI) Blankets

Flexible Insulation (FI) blankets (originally developed as AFRSI) protect regions of the upper surface of each vehicle where moderate heat loads, pressure gradients, and less air flow are encountered. The FI blanket is used where temperatures do not exceed 1,500°F. FI blankets are comprised of quartz fiber batting that is sandwiched between high temperature woven quartz fiber outer fabric and a lower temperature glass inner fabric. The components are stitched together as shown in *Figure 8* using quartz and glass threads in a one-inch square pattern. The plan form size can be up to 30 inches by 30 inches and the thickness varies (with heat load) between 0.41 inch and slightly less than 2 inches. The blanket is bonded directly to the structure using RTV silicone adhesive. Nomex felt ramping, filler bar, and SIP can be used between the FI blanket and structure to allow the installation to fair into adjacent installations. To toughen the outer fabric, the OML surface of the blanket is protected with a ceramic coating. In certain areas, FI blanket requires rewaterproofing to reduce the potential weight increase from absorbed water at launch. This is accomplished by injections of dimethylethoxysilane (DMES) through plastic film on 4-inch centers and covering for 24 hours.



Thread

Section D-D



RTV Transfer Coated Surface on IML



Figure 8 - FI Blanket Construction

Detail E

The fabrication of the FI blanket primarily involves the assembly of its components. The insulative batting is comprised of 6 pcf quartz fiber. The outer fabric is a 0.027 inch thick quartz fiber woven fabric with an aminosilane binder finish. The inner fabric is a 0.009 inch thick S2-glass yard plain woven fabric with a semi-clean finish. The OML thread is 0.029 inch diameter

quartz fiber thread coated with polytetrafluoroethylene (PTFE). The IML thread is 0.020 inch diameter E-glass thread with a liner polyamide coating. The batting is sandwiched between the outer and inner fabrics. The materials are stitched together at 3 to 4 stitches per inch with the two threads interlacing at the IML. The parallel stitch lines are one inch apart in both the length and width directions. The IML fabric and batting are trimmed to a modeled template of the cavity. The OML fabric is folded around the sidewall edges and wrapped around to the IML surface.

The corners are looped stitched with the OML thread. The folded OML fabric is stitched to the blanket using a similar two-thread interlacing stitch technique. The blanket is identified by rubber stamping the part number and order control number (OCN) with liquid bright gold ink. The blanket is waterproofed by the vapor deposition of methyltrimethoxysilane (refer to Section 1.1.4). The part is heat cleaned at 600°F for 2 hours and at 850°F for 4 hours to remove processing aids and oils. A pressure pad consisting of latex foam and Plexiglas is custom made to the particular part. In addition, a 6 inch by 9 inch peel test coupon is fabricated from the identical lots of materials used during the blanket fabrication. The peel test coupon is a process control device that ensures proper adhesion between the blanket IML and structural adhesive. The fabricated blanket, peel test coupon, and pressure pad are delivered for installation.

1.2.1 FI Blanket Installation

The installation of the FI blanket per ML0601-9024 process 501 is depicted in *Figure 9*. The part cavity is pre-cleaned following the removal of the previous part. The cavity is modeled using a template. Following the fabrication of a blanket to the template, the blanket is prefit into the cavity. Ramping or other sub-insulation is installed under pressure using RTV silicone adhesives on a solvent cleaned and primed substrate. The cavity and peel test coupon plate are solvent cleaned, primed, and coated with 0.006 to 0.010 inches of RTV silicone adhesive. The transfer coated surfaces on the sub-insulation are wiped with 1,1,1-trichloroethane and allowed to dry for 2 to 24 hours prior to bonding. The blanket is bonded to the cavity and the peel test coupon is bonded to the plate under 1.5 to 3 psi pressure. Following the cure, the peel test coupon is cut into 1-inch wide strips. A 90° pull test is performed with a force gauge on at least 4 of the strips. The average peel strength of the pulls must be greater than 4 pounds per inch to provide a confidence with the blanket bond.



Figure 9 - FI Blanket Replacement Flow Diagram

The step and gap of the bonded blanket are measured and any large gaps are filled with a FI blanket type gap filler. There are several types of FI blanket type gap fillers. Primarily they are comprised of 0.040-inch thick high-boria content aluminoborosilicate fiber (Nextel) woven fabric, Nextel braided sleeving, Nextel ceramic fiber cord, alumina fiber (Saffil) insulative batting, and ceramic fiber thread. With these materials, there are essentially three types of gap fillers: folded fabric, stuffed sleeving, and fabric-wrapped cord tadpoles (referencing the cross-sectional appearance). The gap fillers are bonded to the blanket sidewall using RTV silicone adhesives. Following the adhesive cure, the gap fillers are stitched to the adjacent blankets using ceramic thread.

To toughen the outer fabric, a C9 ceramic coating is applied to the outer surface of the FI blanket in a two-part process. An 80% Ludox ammonia stabilized colloidal silica solution and 20% isopropyl alcohol precoat mixture is applied and air dried for 4 hours. This precoat

modifies the fabric to promote the adhesion of the topcoat material. The topcoat consists of a mixture of the Ludox ammonia stabilized colloidal silica solution and silica powder that is applied to the blanket and is air dried for 8 hours. The blanket is re-identified using liquid gold bright ink.

1.3 Felt Reusable Surface Insulation

FRSI panels protect most of the upper surface of each vehicle where temperatures are less than 750°F. FRSI is composed of two materials, a heat treated Nomex felt and a vented white silicone elastomer coating. A typical FRSI component is depicted in *Figure 10*. Additional layers of FRSI or Nomex felt ramping can be used between the FRSI and structure to allow the installation to fair into adjacent installations. FRSI does not require post-flight rewaterproofing because the Nomex polymer is hygrophobic by nature and the silicone elastomer coating inhibits water intrusion into the felt.



Figure 10 - FRSI Detail and Replacement Flow Diagram

The Nomex felt is made up from 3 inch long, 2 denier's fine polyamide aramid fibers. The fibers are loaded into a carding machine that combs the tangled fibers into a cross-lapped web. Two webs are placed together and are needle punched. This sewing-like process passes barbed needles through the webs to compact the fibers into a felt pad of the desired properties. The felt is calendared by passing it through rollers to stabilize the thickness. The felt is heat set at 500°F for 30 minutes to provide dimensional stability. The color of the heat set felt is offwhite. This material is used for strain isolator pads (SIP). In all other Nomex felt applications (e.g., FRSI, filler bar, ramping) the felt is heat treated at 700°F for 30 minutes and then at 750°F for 30 minutes to minimize the linear shrinkage at elevated temperatures. The heat treatment darkens the felt to a caramel color.

For FRSI, the heat-treated Nomex felt is transfer coated with a white silicone elastomer. The silicone elastomer is poured and spread to a thickness of 0.006 to 0.008 inch on a screen mesh that was prepared with a parting liquid. The coating is partially cured by air drying for 5 hours. The partially casted coating is coated with additional elastomer to provide a wet layer of coating. The Nomex felt is placed in the coating and is bonded under 2 to 3 psi for 2.5 hours. The part is post cured at 650°F for 15 minutes and air dried for 96 hours.

For all other Nomex felt applications (e.g., SIP, filler bar, ramping, sub-surface FRSI), the felt is placed in a 0.006 to 0.010 inch thick layer of red RTV silicone adhesive. The adhesive is bonded to the felt under 2 to 3 psi until cured.

The installation of FRSI per ML0601-9024 process 401 is the least complex of the three RSI material installations as shown in Figure 10. The FRSI is trimmed to a cavity template. The exposed edges are paint sealed with a white silicone elastomer. The FRSI is bonded under 2 to 3 psi pressure to a solvent cleaned and primed cavity and/or over sub-insulation. The FRSI-to-FRSI joints are sealed with an RTV silicone adhesive, and other interfaces are filled with an RTV silicone adhesive edge member casting. The coating is vented by 0.035 inch holes made on 6 inch centers. FRSI does not require part identification.

1.4 Reinforced Carbon-Carbon

Reinforced carbon-carbon (RCC) is used as a high-temperature aerodynamic structure on the leading edge structural subsystem (LESS) which consists of the nose cap, chin panel, wing leading edge (WLE), and associated expansion seals. In addition, the external tank (ET) forward attach point adjacent structure is protected by an RCC arrowhead component due to the pyrotechnic shock environment of the ET separation mechanism. The RCC material has a maximum use temperature of over 2,960°F and has a density of approximately 103 pcf. The material has a flexural strength of approximately 9,000 psi and a tensile strength of approximately 4,500 psi.

RCC is a structural composite consisting of two discrete carbon-based components, a high-strength substrate and an oxidation protection coating system. The fabrication of RCC is a four-part process as shown in *Figure 11*.



Figure 11 - RCC Fabrication Flow Diagram

The carbon substrate is fabricated from 19 to 38 plies of laid-up phenolic-impregnated graphite fiber cloth autoclave cured at 300°F for 8 hours, rough trimmed, and drilled. The part is

post cured by heating up to 500° F for 7 days. The part is loaded in a graphite retort with calcined coke and is made rigid by converting the phenolic resin to carbon by a 70-hour 1,500°F pyrolysis cycle in an argon atmosphere. The part is designated as "RCC-0" and has a flexural strength of approximately 3,000 psi. The part is then densified by vacuum impregnation of furfural alcohol and conversion to carbon by pyrolysis. The subsequent pyrolyses are performed by a 2 hour 300°F autoclave cure followed by a 400°F post cure for 32 hours. The furfural alcohol vacuum impregnation and pyrolysis cycles are repeated three times. After the final pyrolysis, the part is designated "RCC-3" and has a significantly stiffer flexural strength of approximately 18,000 psi. The final machining of the part is performed. The pure carbon substrate is subject to oxidation at temperatures over 700°F, well below the service temperature of the component. Therefore, an oxidation protection coating is required. The term "coating" is actually a misnomer as the outer surfaces (0.020 to 0.040 inch) of the carbon component are converted to silicon carbide by a diffusion reaction. The conversion process is accomplished by packing the component into a mix of constituent powders (60% silicon carbide, 30% silicon, and 10% alumina) in a graphite retort and is subjected to a 16 hour heating cycle which includes a 600°F drying cycle and a diffusion coating cycle with temperatures up to 3,000°F in an argon atmosphere. The carbon substrate and silicon carbide materials have a thermal expansion mismatch which results in the formation of very small craze cracks in the silicon carbide layer as the silicon carbide contracts more than the carbon substrate during the cool down period. To provide further protection, the RCC part is vacuum impregnated with tetraethyl orthosilicate (TEOS) and oven cured at 225°F for 45 minutes. The TEOS impregnation and heat curing is repeated four times with the fifth oven cure at 225°F for 2 hours. The part is heat cured at 400°F

for 30 minutes and 600°F for 6 hours. The heat cures result in the formation of a protective layer of silicon dioxide residue.

The final fabrication step is to apply Type A sealant to fill any porosity or craze cracks on the RCC part. The Type A sealant is a mixture of silicon carbide powder and a sodium silicate water glass. The mixture is prepared and is brushed on the part. The part is then air dried for 16 hours and heat cured at 200°F for 2 hours, 400°F for 2 hours, and 600°F for 4 hours. The application and subsequent curing is repeated. Once the fabrication is complete, the part is ready for installation.

The LESS is made up of two distinct entities, the nose area and the wing leading edge, as shown in *Figure 12* and *Figure 13*, respectively. The nose area is protected by the RCC nose cap, the chin panel, and nine associated expansion and tee seals. The wing leading edge is protected by 44 RCC panels, 42 RCC tee seals, and 2 angle expansion seals. The parts are mechanically attached to the aluminum forward bulkhead or wing spar using inconel 718 and A-286 fittings on floating joints. The floating assembly is used to prevent excessive loading and to seal the RCC cavity from hot plasma flow. The attachment of the nose cap and chin panel seals allows for circumferential, fore, and aft movement about the nose cap periphery. The angle (located forward of panel 1) and tee seals on the wing leading edge allow for lateral motion and thermal expansion differences between the RCC and wing. To further prevent the flow of hot gas from entering the RCC cavities, alumina-stuffed aluminoborosilicate (Nextel) gap fillers are used on the lower surface between the RCC and HRSI tile interfaces. The open interface gap on the upper surface between the RCC and HRSI tiles allows for venting of the RCC cavity in the thermally benign regions of the LESS.



Figure 12 - LESS Nose Area RCC Components



Figure 13 - LESS Wing Leading Edge RCC Components

The RCC material promotes the internal cross radiation from the hot stagnation region at the apex to cooler areas. This cross radiation reduces the temperatures at the apex and increases the temperatures of the cooler regions which, in turn, reduce the thermal gradients around the component. This cross radiation also directs heat back to the structure. Therefore, the structure must be protected by the utilization of backing insulation. The nose cap and chin panel use an uncoated flexible insulation blanket fabricated from aluminoborosilicate fiber fabric (Nextel) and alumina insulation (Saffil) or alumina silica chromia (Cerachrome) to protect the structure. In addition, high-temperature reusable surface insulation (HRSI) tiles are bonded to the forward bulkhead to offer additional thermal protection behind the nose cap. An uncoated FI blanket is used as the insulation beneath the arrowhead. The radiation from the wing leading edge RCC to the wing spar is protected by 0.030 inch thick inconel foil covered Cerachrome batting known as Incoflex insulators. Although the intent of the backing insulation is to protect the structure, it also retards the internal RCC cross radiation and subsequently retards the cooling rate of the RCC lugs adjacent to the backing insulation. This prolonged heating contributes to the undesirable oxidation rate of the RCC which, in turn, reduces the mission life of the component.

1.5 Gap Fillers

Gap fillers are used in areas to restrict the flow of hot gas into the gaps of TPS components. The types and applications of the various types of gap fillers are shown in *Figure 14*. The predominant gap filler types that are used are the pillow or pad type and the Ames type.



Figure 14 - Tile-to-Tile Gap Fillers

The pillow fabric gap fillers are usually installed to completely fill their intended gaps. The basic pillow gap filler is fabricated from a template (depicting the contour, height, and width required) of the gap with specific thickness requirements recorded on the Mylar. The gap filler fabrication begins with trimming a 0.001-inch thick sheet of Inconel 601 alloy to the shape of the gap to be filled. The aluminoborosilicate fiber (Nextel) fabric is folded over the inconel, and the fabric is stuffed with an alumina fiber (Saffil) batting to obtain the desired thickness. The gap filler is stitched with Nextel thread. The tail of the gap filler is stiffened with RTV silicone adhesive. The other types of stitched gap fillers are derivations of the basic pillow type. The derivations include the use of Nextel ceramic fiber braided sleeving. The sleeving can be added to the exterior or interior of the folded area of the gap filler fabric.

The majority of gap fillers are installed following the installation of RSI tiles. The gap filler is bonded to the underlying filler bar or tile sidewall with RTV silicone adhesive. Following the cure of the adhesive, the gap filler is friction tested to ensure the proper compression within the gap and to validate the integrity of the gap filler bond. Pillow and pad-type gap fillers are coated with a high emissivity ceramic coating in a two-part application procedure similar to that of FI blankets. A 85% Ludox ammonia stabilized colloidal silica solution, 12% isopropyl alcohol, and 3% silicon carbide powder precoat mixture is applied and air dried for 4 hours. This precoat modifies the fabric to promote the adhesion of the topcoat material. The topcoat consists of a mixture of the Ludox ammonia stabilized colloidal silica solution, silica powder, silicon carbide powder that is applied to the exposed area of the gap filler and is air dried for 8 hours.

There are three varieties of Ames gap fillers comprised of two fabric types and two coating types. The fabric is available in a non-vacuum baked and vacuum baked condition. The non-vacuum baked fabric can be coated with black RTV for upper surface use and ceramic coating for lower surface use. The vacuum baked variety can only be fabricated with the black RTV coating for upper surface use.

The Ames gap filler is nominally 0.020 inch thick and is cut to fit a gap Mylar. Up to 6 layers of Ames gap fillers are installed to fill a gap partially or completely. A Mylar is made of the gap which duplicates the length, width, and contour of the gap with gap measurements recorded in the corresponding locations on the Mylar. The gap filler is prefit and pull test loops are installed. The gap filler is installed by RTV bonding onto a primed surface, and the bond is verified by pulling on test loops after the adhesive cure.

1.6 Thermal Barriers

Thermal barriers are used around penetrations and in the closeout areas between the major components of the orbiter. The primary purpose is to restrict hot gas flow to the underlying cavity or structure. The locations of the orbiter thermal barriers (and aerothermal seals, Section 1.7) are shown in *Figure 15*.



Aerothermal Seals		
A	Wing/Elevon	
В	Aft Fuselage/Body Rap	
С	Vertical Stabilizer/Rudder Speed Brake	
D	Payload Bay Door Expansion Joints	
E	Payload Bay Door Hinge Covers	

Figure 15 - Thermal Barrier and Aerothermal Seal Locations

The majority of thermal barriers are constructed from spring tube, insulative batting,

sleeving, and ceramic fabric. The spring tube is a tubular inconel wire mesh. The part is

inserted into aluminoborosilicate fiber (Nextel) braided sleeving. The thermal barrier is then covered with a Nextel ceramic fiber fabric outer cover. The thermal barrier is bonded by its ceramic fabric tail to its intended cavity (for adhesive bonded types), attached to the structure by the use of hardware (for mechanically attached types), or attached to a carrier plate (for mechanically attached carrier panel types). *Figure 16* depicts the mechanically attached carrier panel type thermal barrier installed around the periphery of the main landing gear doors.



Figure 16 - Main Landing Gear Door Thermal Barrier Detail

Thermal barriers are installed per specific processes for the particular design. They are usually bonded under pressure to a solvent cleaned and primed structural substrate with RTV silicone adhesive. The outer thermal barriers in the thermally extreme nose landing gear door area are bonded to the peripheral HRSI tile sidewalls and RCC surfaces with a ceramic adhesive. The ceramic adhesive is a two component mixture. The first component is a 75% deionized water and 25% Ludox ammonia stabilized colloidal silica solution. The second component is a ceramic adhesive powder. The thermal barriers on the main landing gear and external tank doors are bonded to a solvent cleaned and primed carrier panel using RTV silicone adhesive. The carrier panel is clipped into a retaining fixture affixed to the orbiter structure. The thermal barriers around the nozzles of the reaction control system (RCS) thrusters are attached to the structure using fasteners.

Following installation the thermal barrier outer fabric is coated. The coating is made of a polyethylene or a black RTV silicone adhesive. The coatings provide improved thermal performance and durability.

1.7 Aerothermal Seals

Aerothermal seals are used to restrict hot gas flow into the control surface cavities and payload bay door areas. *Figure 15* depicts the locations of the aerothermal seals.

The wing trailing edge/elevon leading edge (i.e., the elevon cove) and the aft fuselage trailing edge/body flap leading edge (i.e., the body flap cove) are thermal seals. *Figure 17* depicts the aerothermal seal in the elevon cove region. The primary seal in this region is the span wise polyimide seal which contacts the elevon rub tube. This seal requires a precise fit against the rub tube to limit the flow into the cavity during control surface movement. Within the cavity, there are heat sinks and additional insulative material to increase the thermal mass and reduce structural thermal gradients. At the inboard and outboard ends of the control surfaces, there are spring loaded columbium seals to prevent hot flow from entering the cavity and potentially overheating the underlying structure and mechanisms. This spring loaded seal allows for the inboard and outboard floating of the elevon due to thermal expansion mismatches between the wing and elevon. The upper surface of the elevon cove is sealed with inconel flipper doors. These flipper doors are hinged on the wing trailing edge and move in concert with the elevon to ensure a proper seal with the rub panels on the upper elevon. The exposed metallic surface is coated with white paint to optimize the thermal emissivity of the part.



Figure 17 - Elevon Cove Aerothermal Seal Detail

The payload bay door area is protected by two types of aerothermal seals as shown in *Figure 18.* The expansion joints are sealed by environmental bulb seals. These FEP Teflon seals are protected during reentry by a quartz fibrous pile thermal barrier. The sealing surfaces are coated with a fluorinated grease to prohibit water intrusion into the payload bay. The payload bay door hinge area is protected by a spring loaded inconel 718 cover assembly. This assembly is used on the first six hinges on OV-102 (Columbia) and the first ten hinges on OV- 103 (Discovery) and subsequent orbiters (Atlantis and Endeavour). The design allows for floating as the spring loaded piston is driven inward towards the center clevis cover. This floating design allows for fore and aft movement of the graphite epoxy composite payload bay doors for the

thermal expansion mismatch with the aluminum alloy midfuselage. The exposed surfaces of the hinge cover are coated with the high emissivity Pyromark coating.



Figure 18 - Payload Bay Door Aerothermal Seals

1.8 Windows

There are eleven windows on the orbiter to provide visibility for mission operations. There are six forward windows, two overhead windows, two aft flight deck windows, and one crew hatch window. The window locations and their designations are shown in *Figure 19*. The forward, overhead, and crew hatch windows consist of three panes of glass held in a pressure sealed retainer. The outermost pane is attached to the forward fuselage structure and the inner two panes are attached to the crew module. The aft flight deck windows have only two panes of glass attached to the crew module. The outermost pane is the only window component of the thermal protection system. The window installation configuration is shown in *Figure 19*.

The innermost pane is the pressure pane. It is fabricated from an aluminosilicate glass which is tempered to provide the strength required to withstand the crew compartment on-orbit pressure differential. The pressure pane, along with the thermal pane, is designed to withstand a pressure of 8,600 psi at 240°F. The outer surface of this pane is coated with an infrared reflective coating. This pane is 0.625 inch thick on the forward windows, 0.450 inch thick on the overhead windows, 0.300 inch thick on the aft flight deck windows, and 0.250 inch thick on the crew hatch window.

The center pane is the redundant pane. It is fabricated from a low-expansion fused silica glass. This uncoated pane is 1.300 inch thick on the forward windows, 0.450 inch thick on the overhead windows, 0.300 inch thick on the aft flight deck windows, and 0.500 inch thick on the crew hatch window. The outermost pane is the thermal pane. It is fabricated from the same fused silica glass as the redundant pane. This pane is designed to withstand the same pressure as the pressure pane. The interior of this pane is coated with a high-efficiency anti-reflective

coating to improve light transmission. This pane is 0.625 inch thick on the forward windows, 0.680 inch thick on the overhead windows, and 0.300 inch thick on the crew hatch window.



Figure 19 - Orbiter Window Locations and Installation Detail

2.0 ORBITER VEHICLE 105, ENDEAVOUR, TILE SUBNOMINAL BOND ISSUES

During summer 2003, structures work along the wing/fuselage mate rivet line Orbiter Vehicle 105, Endeavor forced the removal of several tiles for rivet inspection and replacement. During the removals several tiles were identified to have a subonominal bond between the tile and the SIP. This was an unusual subnominal bond for the TPS as it had never been identified in the past. *Figure 20* shows the location along the wing/fuselage mate line that tiles were removed.



Figure 20 - 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 (Refer to *Figure 21*). 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 shown in *Figure 22*. An adhesive peel, explained in Appendix C, is considered a subnominal bond condition, and is referred to as a subnominal SIP/IML adhesive bond. A nominal SIP to tile bond should have a coat of red RTV on the surface of the tile once the SIP is removed as shown in *Figure 23*.



Figure 21 - Cross Section of Tile Adhesion with Tensile Strengths



Figure 22 - OV-105 Subnominal Bond Condition



Figure 23 - Nominal SIP to Tile Bond Condition

2.1 Testing 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 (Appendix A). 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.

2.2 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 (Refer to *Figure 24*). No engineering investigation occurred.



Figure 24 - 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, densification, and SIP bond logs were reviewed for commonality. Additional subsets of tiles 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 as shown in *Table 2*

Table 2 - SIP/IML Interface Processes

Process	Description
Densification	Slurry application to the tile IML
Waterproofing	Tile waterproofing performed prior to SIP bond
SIP Bond	SIP application to the tile IML with RTV
2.2 Contamination Testing and Analysis

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 molecules and then detect and collect data showing the quantity of the various molecules collected.

2.3 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. Appendix D analyzes the results of the review.

2.4 Bond Verification 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 as shown in *Figure 25*. 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 25 - Bond Verification Check Setup

2.5 SIP Peel Tests

As there is no RTV adhesive peel requirement, this was an engineering evaluation only: a peel value greater than $4\frac{lb}{in}$ 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 as shown in *Figure 26*.



Figure 26 - SIP Peel Test Being Performed on Subnominal Bond Tile

2.6 Staged Tests and Procedures

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 with different factors and levels as shown in *Table 3*.

Factors	Levels	
	1 - Double nominal amount	
RTV catalyst quantity	2 - Nominal amount	
	3 - Half of nominal amount	
RTV quantity applied to tile IML	1 - Nominal amount	
	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	
	1 - contact pressure	
Pressure (force) applied	2 - nominal pressure (1.5 psi)	
	3 - over pressure (3.5 psi)	

Table 3 - Factors and Levels used for Testing

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.

3.0 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. The comparison of tensile strength of the tile against the flight load, the BV load and the peel strength can be seen in *Figure 27*. A typical peel strength for a nominal tile bond is greater than $4\frac{lb}{in}$, anomalous tiles revealed a peel strength as low as $0.5\frac{lb}{in}$. A comparison of

66

BV strength against the peel strength of the SIP and also the flight stresses that the tile sees can be found in *Figure 28*. Full peel strength results are in Appendix B.



Figure 27 - Comparison of Tensile Strength in Tile during BV, Flight Loads, and Peel Strength



Figure 28 - Peel Strength of SIP against BV Strength and Flight Stress

3.1 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. The graphical results of these tests are found in *Figure 29*. *Figure 30*, *Figure 31*, *Figure 32*, *Figure 33*, and *Figure 34* contain the individual results for each of the tests which were ran and included in *Figure 29*.



Figure 29 - Results of Changing Process Variables to SIP Bond Strength



Figure 30 - Catalyst Weight vs. Average Peel Strength



Figure 31 - Amount of RTV Applied vs. Average Peel Strength



Figure 32 - RTV Application Catalyst Drop Time vs. Average Peel Strength



Figure 33 - Pressure Application vs. Average Peel Strength



Figure 34 - Pressure vs. Peel Strength

The time 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 Spray Starch and MS-143 Mold Release Agent as shown in *Figure 35*, *Figure 36*, and *Figure 37*. Additionally, as the amount of contaminants added increased, the peel strength decreased.





Figure 35 - Peel Test Results for Process Variations



Figure 36 - Peel Test Results IML Contaminated with Krylon Spray Starch



Figure 37 - Peel Test Results IML Contaminated with MS-143 Mold Release Agent

4.0 SUMMARY OF TEST 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; densification workmanship; densification process deficiency; factory waterproofing workmanship; SIP bond process contamination; vehicle location; and age issues. (The reasoning behind the elimination of all other factors in discussed in the Historical Document Review analysis in Appendix D.) The caused listed above will be discussed in detail in the following chapters.

4.1 **Oven Pump Malfunction**

An option that can be eliminated as a possible cause of a subnominal SIP/IML adhesive bond is that the oven pumps 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 38* contains a photo of a tile with no waterproofing installed. As can be seen in the photo the subnominal condition is not similar to the one identified on OV-105.



Figure 38 - Peel without Waterproofing; Average Peel 13.5 lb/in

4.2 Densification Material Anomaly

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.

4.3 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 39 - SIP Peel Test without Silane Average Peel 6 lb/in



Figure 40 - Normal Peel Test Average Peel 22 lb/in

4.4 SIP Bond Process Environmental Condition

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. One concern with the environmental conditions is that the humidity is required to be at a higher level in order fore the RTV to cure properly. When the humidity is low the RTV cures very slowly. If the humidity was high then the RTV cures faster. In that situation there was a possibility that the RTV cured prior to being applied to the tile. The time span during which the SIP/IML bond could have been affected had the conditions as shown in *Table 4*.

Table 4 - Weather Conditions at Palmdale at Tile Installation

	Min	Max
RH Level	14.3	100
Тетр	-11.0°C	39.9°C
Precipitation	0.0in	0.65in

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).

4.5 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. The SIP peel test which was performed with minimal catalyst can be seen in *Figure 41*. This peel test can be compared to the nominal peel photo in *Figure 40*.



Figure 41 - SIP Peel Test with 0.25g (minimal) Catalyst

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. Several photos showing the different amounts of RTV application can be seen in *Figure 42* and **Error! Reference source not found.**.



Figure 42 - SIP Peel Test with 3.61g of RTV Applied



Figure 43 - SIP Peel Test with 0.78g of RTV Applied

4.6 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.

4.7 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. The SIP peel test which was performed with the Krylon 1201 spray starch can be seen in *Figure 44*. The SIP peel test which was performed with MS-143 mold release agent is identified in *Figure 45*.



Figure 44 - SIP Peel Test with Krylon 1201 Spray Starch



Figure 45 - SIP Peel Test with MS-143 Mold Release Agent

4.8 Vehicle Location

Data research showed that all of the SIP to tile subnominal bonds were Palmdale tile 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.

4.9 Age Issues

Analysis and document review has neither eliminated nor advanced the possibility that the SIP bond degraded over time. The chart identified in *Figure 46* compares the Shore A hardness of a typical tile removal against the Shore A hardness of the RTV removed from the OV-105 subnominal bond tiles. The Materials and Processes of TPS had determined during the early parts of the program that anything that shows a Shore A hardness of below 30 is a cause for concern in the TPS system. As seen in the chart all of the samples that had subnominal bonds had Shore A hardness near 55.



Figure 46 - Shore A Hardness of Typical RTV vs Subnominal Bond RTV

5.0 **RECOMMENDATIONS**

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

5.1 Testing Process

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. The results of the peel tests at the different labs can be seen in



Figure 47 - Comparison of Lab Data from KSC and Huntington Beach

5.2 Brush Cleaning

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.

5.3 Krylon 1201 and MS-143

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.

5.4 Future Monitoring

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.

APPENDIX A: SIP BOND PROCESS VARIABLES

Catalyst	RTV			Pressure
Weight	applied	Pressure	RTV Application Time	Application Time
Nominal	Nominal	Nominal	Late	Late
Nominal	Nominal	Too High	Late	Late
Nominal	Nominal	Too Low	Nominal	Nominal
Nominal	Too Little	Nominal	Nominal	Late
Nominal	Too Little	Too High	Nominal	Late
Nominal	Too Little	Too Low	Late	Late
Too Little	Nominal	Too Low	Late	Nominal
Too Little	Too Little	Too Low	Nominal	Nominal
Too Much	Nominal	Nominal	Late	Late
Too Much	Nominal	Too High	Late	Late
Too Much	Nominal	Too Low	Late	Nominal
Too Much	Nominal	Too Low	Nominal	Nominal
Too Much	Too Little	Nominal	Nominal	Late
Too Much	Too Little	Too High	Nominal	Late
Too Much	Too Little	Too Low	Nominal	Nominal
Too Much	Too Little	Too Low	Late	Late
Nominal	Nominal	Nominal	Late placing SIP onto tile IML	Nominal

				RTV	Pressure
	Catalyst	RTV		Application	Application
Tile	Weight	applied	Pressure	Time	Time
-001	Too Much	Too Little	Too Low	Nominal	Nominal
-002	Too Much	Too Little	Too Low	Nominal	Nominal
-003	Too Little	Too Little	Too Low	Nominal	Nominal
-004	Too Little	Too Little	Too Low	Nominal	Nominal
-005	Too Little	Too Little	Too Low	Nominal	Nominal
-006	Too Much	Too Little	Too Low	Nominal	Nominal
-007	Too Much	Nominal	Too Low	Late	Nominal
-008	Too Much	Nominal	Too Low	Late	Nominal
-009	Too Little	Nominal	Too Low	Late	Nominal
-010	Too Little	Nominal	Too Low	Late	Nominal
-011	Too Little	Nominal	Too Low	Late	Nominal
-012	Too Much	Nominal	Too Low	Late	Nominal
-013	Too Much	Too Little	Too Low	Late	Late
-014	Nominal	Too Little	Too Low	Late	Late
-015	Too Much	Too Little	Too Low	Late	Late
-016	Nominal	Too Little	Too Low	Late	Late
-017	Nominal	Too Little	Too Low	Late	Late
-018	Too Much	Too Little	Too Low	Late	Late
-019	Nominal	Too Little	Nominal	Nominal	Late

				RTV	Pressure
	Catalyst	RTV		Application	Application
Tile	Weight	applied	Pressure	Time	Time
-020	Too Much	Too Little	Nominal	Nominal	Late
-021	Nominal	Too Little	Nominal	Nominal	Late
-022	Nominal	Too Little	Nominal	Nominal	Late
-023	Too Much	Too Little	Nominal	Nominal	Late
-024	Too Much	Too Little	Nominal	Nominal	Late
-025	Nominal	Nominal	Nominal	Late	Late
-026	Too Much	Nominal	Nominal	Late	Late
-027	Nominal	Nominal	Nominal	Late	Late
-028	Nominal	Nominal	Nominal	Late	Late
-029	Too Much	Nominal	Nominal	Late	Late
-030	Too Much	Nominal	Nominal	Late	Late
-031	Nominal	Too Little	Too High	Nominal	Late
-032	Too Much	Too Little	Too High	Nominal	Late
-033	Too Much	Too Little	Too High	Nominal	Late
-034	Too Much	Too Little	Too High	Nominal	Late
-035	Nominal	Too Little	Too High	Nominal	Late
-036	Nominal	Too Little	Too High	Nominal	Late
-037	Nominal	Nominal	Too High	Late	Late
-038	Too Much	Nominal	Too High	Late	Late
				RTV	Pressure
------	----------	---------	----------	---------------	-------------
	Catalyst	RTV		Application	Application
Tile	Weight	applied	Pressure	Time	Time
-039	Nominal	Nominal	Too High	Late	Late
-040	Too Much	Nominal	Too High	Late	Late
-041	Nominal	Nominal	Too High	Late	Late
-042	Too Much	Nominal	Too High	Late	Late
-043	Too Much	Nominal	Too Low	Nominal	Nominal
-044	Too Much	Nominal	Too Low	Nominal	Nominal
-045	Nominal	Nominal	Too Low	Nominal	Nominal
-046	Nominal	Nominal	Too Low	Nominal	Nominal
-047	Nominal	Nominal	Too Low	Nominal	Nominal
-048	Too Much	Nominal	Too Low	Nominal	Nominal
				Late placing	
				SIP onto tile	
-049	Nominal	Nominal	Nominal	IML	Nominal
				Late placing	
				SIP onto tile	
-050	Nominal	Nominal	Nominal	IML	Nominal
				Late placing	
				SIP onto tile	
-051	Nominal	Nominal	Nominal	IML	Nominal
-052	Nominal	Nominal	Nominal	Late placing	Nominal

				RTV	Pressure
	Catalyst	RTV		Application	Application
Tile	Weight	applied	Pressure	Time	Time
				SIP onto tile	
				IML	
				Late placing	
				SIP onto tile	
-053	Nominal	Nominal	Nominal	IML	Nominal
				Late placing	
				SIP onto tile	
-054	Nominal	Nominal	Nominal	IML	Nominal

No waterproofing
1/2 kit acetic acid and 1/2 kit silane
no silane
no acetic acid
1/4 kit acetic acid and 3/4 kit silane
3/4 kit acetic acid and 1/4 kit silane
heat clean after waterproofing
Normal waterproofing - no process
variation

Table 6 - Waterproofing Process Variation

Table 7- Contamination Prior to/During SIP Bond

IML contaminated with FC724
IML contaminated with TCA and MEK
IML contaminated with Tri-Flo lubricant
IML contaminated with Krylon 1301 spray starch
IML contaminated with MS-143 Mold Release
Agent

L

APPENDIX B: TEST RESULTS

Anomalous Bonds:							
	Peel						
Part Number	Strength	BV Stress	Flight				
	(lb/in width)	(PSI)	Stresses				
-069	N/A	N/A	4.00				
-070	0.5	6*	4.30				
-071	0.5	14.8	7.61				
-072	<0.5	15.8	4.30				
-084	N/A	N/A	4.16				
-089	0.5-2.0	7.7*	8.25				
-091	1.0-2.0	*	4.16				
-094	N/A	10	8.70				
-095	1.5	10	8.70				
-096	1.5	10	8.74				
-098	N/A	N/A	8.70				
-099	N/A	N/A	7.81				
-101	N/A	N/A	7.81				
-103	0.5	10	4.70				
-106	N/A	20	6.70				
-193	N/A	18.8	8.25				
-200	N/A	N/A	8.74				
-202	N/A	16.2	8.70				
-204	N/A	17.7	8.70				
-205	N/A	9.6	7.81				
-210	3	20	6.70				

Table 8 - V070-190002 Tile Subnominal Bonds

-139	0.5	18.6	3.61				
-152	1.5	19.7	3.61				
-146	1	10	4.85				
-147	N/A	10	8.96				
-148	N/A	10	4.36				
-158	N/A	10	4.85				
-329	N/A	N/A	3.99				
* Failure in tile coating due to star cracks							

				Time				Visual
			Time	Between				
			Between	RTV				
	Catalyst	Amount	RTV	Application		Avg.	Avg.	
	Weight	of RTV	Application	and		Min.	Max.	
	(g/100	applied	and Catalyst	Pressure	Pressure	Peel	Peel	
Tile	g)	(g/in ²)	Drop	Application	(psi)	(lb/in)	(lb/in)	
-001	1.00	0.04	0:20	0:22	contact	15. 2	20	
-002	1.00	0.04	0:22	0:24	contact	22.2	27	
-003	0.25	0.04	0:06	0:08	contact	14.8	19.6	
-004	0.25	0.04	0:09	0:10	contact	14.6	19	
-005	0.25	0.04	0:11	0:12	contact	22.6	27.2	11
-006	1.00	0.04	0:25	0:27	contact	17.4	19.8	
-007	0.93	0.09	0:54	0:56	contact	15	21.6	
-008	0.98	0.09	0:57	0:58	contact	16.5	24	AL.
-009	1.04	0.10	1:31	1:33	contact	19.2	24.8	X
-010	1.09	0.09	1:35	1:37	contact	19.2	24.6	

Table 9- MISC-794-480 Test Results

				Time				Visual
			Time	Between				
			Between	RTV				
	Catalyst	Amount	RTV	Application		Avg.	Avg.	
	Weight	of RTV	Application	and		Min.	Max.	
	(g/100	applied	and Catalyst	Pressure	Pressure	Peel	Peel	
Tile	g)	(g/in ²)	Drop	Application	(psi)	(lb/in)	(lb/in)	
-011	1.14	0.10	1:37	1:39	contact	18	24.8	
-012	1.20	0.10	1:01	1:03	contact	17.4	20.4	
-013	1.00	0.03	1:03	1:05	contact	14	18.6	
-014	0.50	0.04	1:40	1:42	contact	16.4	21	New York
-015	1.00	0.03	1:07	1:09	contact	18	24.2	
-016	0.50	0.03	1:44	1:46	contact	19	24.2	
-017	0.50	0.03	1:48	1:50	contact	15.4	20.2	R
-018	1.00	0.04	1:10	1:12	contact	10.6	16.2	
-019	0.50	0.03	0:47	1:06	1.5	10.6	15.2	
-020	1.00	0.03	0:32	1:01	1.5	14.4	21.4	
-021	0.50	0.03	0:54	1:06	1.5	9	14.2	ALL.
-022	0.50	0.03	0:57	1:06	1.5	9	15.2	

				Time				Visual
			Time	Between				
			Between	RTV				
	Catalyst	Amount	RTV	Application		Avg.	Avg.	
	Weight	of RTV	Application	and		Min.	Max.	
	(g/100	applied	and Catalyst	Pressure	Pressure	Peel	Peel	
Tile	g)	(g/in ²)	Drop	Application	(psi)	(lb/in)	(lb/in)	
-023	1.00	0.03	0:35	1:01	1.5	6.8	13.6	
-024	1.00	0.03	0:37	1:01	1.5	11.4	17.6	
-025	0.50	0.10	1:53	2:28	1.5	7.2	15.4	No.
-026	1.00	0.11	1:13	2:05	1.5	8.4	15.2	
-027	0.50	0.11	1:46	2:28	1.5	10.2	18.4	
-028	0.50	0.11	1:52	2:28	1.5	10.2	19	
-029	1.00	0.11	1:17	2:05	1.5	9.6	14.8	
-030	1.00	0.10	1:19	2:05	1.5	9	17	
-031	0.50	0.03	0:31	1:32	3.5	9	15.4	
-032	1.00	0.03	0:23	1:14	3.5	10.8	15.6	
-033	1.00	0.03	0:26	1:14	3.5	9.6	15.6	
-034	1.00	0.03	0:29	1:14	3.5	10.8	16	

				Time				Visual
			Time	Between				
			Between	RTV				
	Catalyst	Amount	RTV	Application		Avg.	Avg.	
	Weight	of RTV	Application	and		Min.	Max.	
	(g/100	applied	and Catalyst	Pressure	Pressure	Peel	Peel	
Tile	g)	(g/in ²)	Drop	Application	(psi)	(lb/in)	(lb/in)	
-035	0.50	0.03	0:33	1:32	3.5	10.4	19.6	M
-036	0.50	0.03	0:35	1:32	3.5	11.4	18.4	W
-037	0.50	0.11	1:30	2:12	3.5	11.2	19.2	
-038	1.00	0.10	0:34	1:11	3.5	7.8	17.6	
-039	0.50	0.10	1:38	2:12	3.5	9.6	18	
-040	1.00	0.09	0:59	1:11	3.5	10.2	18.4	
-041	0.50	0.11	2:04	2:12	3.5	7.2	11.8	
-042	1.00	0.11	1:04	1:11	3.5	13.2	18	
-043	1.00	0.10	0:27	0:29	contact	10.2	17.6	
-044	1.00	0.10	0:29	0:31	contact	9	15.2	
-045	0.50	0.10	0:14	0:16	contact	13.8	22.4	
-046	0.50	0.10	0:16	0:18	contact	10.8	17.8	

				Time				Visual
			Time	Between				
			Between	RTV				
	Catalyst	Amount	RTV	Application		Avg.	Avg.	
	Weight	of RTV	Application	and		Min.	Max.	
	(g/100	applied	and Catalyst	Pressure	Pressure	Peel	Peel	
Tile	g)	(g/in ²)	Drop	Application	(psi)	(lb/in)	(lb/in)	
-047	0.50	0.10	0:18	0:20	contact	12	18	4
-048	1.00	0.11	0:31	0:33	contact	13.4	20.6	
-049	0.50	0.03	0:23	1:33	1.5	15.6	20.2	1
-050	0.50	0.04	0:25	1:31	1.5	20.8	23.6	41
-051	0.50	0.03	0:27	1:29	1.5	12.8	19.6	5
-052	0.50	0.03	0:29	2:39	1.5	15.6	19.2	5.3
-053	0.50	0.03	0:31	2:37	1.5	7.6	19.2	重
-054	0.50	0.04	0:33	2:35	1.5	17.8	22.6	and the second s

		Avg. Min.	Avg. Max.	Visual
Tile	Process Variation	Peel (lb/in)	Peel (lb/in)	
-001	No waterproofing	9.0	18.6	
-002	No waterproofing	10.2	21.2	
-003	No waterproofing	7.2	22.8	
-006	1/2 kit acetic acid and 1/2 kit silane	14.4	21.8	
-007	1/2 kit acetic acid and 1/2 kit silane	11.4	17.4	in the
-008	1/2 kit acetic acid and 1/2 kit silane	14.2	22.2	No.
-011	no silane	3.0	9.0	
-012	no silane	9.0	15.8	
-013	no silane	6.2	11.4	

Table 10 - MISC-794-479 Test Results

		Avg. Min.	Avg. Max.	Visual
Tile	Process Variation	Peel (lb/in)	Peel (lb/in)	
				No.
-016	no acetic acid	8.4	13.4	Mental 1
-017	no acetic acid	8.4	16.0	
				- 1 HA
-018	no acetic acid	12.0	18.6	Contraction of the second
				53.
-021	1/4 kit acetic acid and 3/4 kit silane	12.6	20.4	1
				Jun -
-022	1/4 kit acetic acid and 3/4 kit silane	15.2	18.6	
				111
-023	1/4 kit acetic acid and 3/4 kit silane	15.8	19.4	
				AMA A
-026	3/4 kit acetic acid and 1/4 kit silane	15.2	19.8	
				123
-027	3/4 kit acetic acid and 1/4 kit silane	12.0	17.4	ME .
				12th
-028	3/4 kit acetic acid and 1/4 kit silane	12.6	17.4	All and the second second
				AR. Y
-031	heat clean after waterproofing	17.0	21.2	ALL AL

		Avg. Min.	Avg. Max.	Visual
Tile	Process Variation	Peel (lb/in)	Peel (lb/in)	
-032	heat clean after waterproofing	15.0	24.0	PAGE
-033	heat clean after waterproofing	12.8	19.5	A CONTRACT
				441
-036	Normal waterproofing - no process variation	18.6	26.2	ALC: NO.
				- m \
-037	Normal waterproofing - no process variation	17.2	23.0	Acres 1
				144
-038	Normal waterproofing - no process variation	15.8	19.4	and the second
-048	IML contaminated with FC724	12.2	17.2	
-049	IML contaminated with FC724	13.8	19.6	deh
				A.
-050	IML contaminated with FC724	14.4	19.8	
-051	IML contaminated with FC724	13.8	16.0	
-055	IML contaminated with TCA and MEK	9.2	12.0	

		Avg. Min.	Avg. Max.	Visual
Tile	Process Variation	Peel (lb/in)	Peel (lb/in)	
-056	IML contaminated with TCA and MEK	10.2	12.4	
-057	IML contaminated with TCA and MEK	9.6	15.2	
-058	IML contaminated with TCA and MEK	13.4	16.8	
-071	IML contaminated with Tri-Flo lubricant	9.6	16.8	
-072	IML contaminated with Tri-Flo lubricant	4.6	7.8	
-073	IML contaminated with Tri-Flo lubricant	7.8	13.0	
-076	IML contaminated with Krylon spray starch	0.2	0.3	
-077	IML contaminated with Krylon spray starch	0.5	0.5	
-078	IML contaminated with Krylon spray starch	4.0	6.2	Real of the

	Contamination		Avg. Min.	Avg. Max.	Visual
Tile	Amount (g)	Process Variation	Peel (lb/in)	Peel (lb/in)	
		IML contaminated with Krylon			
-001	0	spray starch	11.2	14.0	11 112
		IML contaminated with Krylon			R
-002	0	spray starch	7.0	9.4	- <u>6</u>
		IML contaminated with Krylon			32
-003	0	spray starch	8.0	14.8	2
		IML contaminated with Krylon			N/A
-004		spray starch	НВ	HB	
		IML contaminated with Krylon			
-005	0.21	spray starch	0.9	2.9	
		IML contaminated with Krylon			(11)
-006	0.19	spray starch	0.3	1.1	1224
		IML contaminated with Krylon			they
-007	0.19	spray starch	0.4	1.5	471
		IML contaminated with Krylon			N/A
-008		spray starch	НВ	HB	
		IML contaminated with Krylon			1000
-009	0.54	spray starch	0.1	0.1	
		IML contaminated with Krylon			(11)
-010	0.62	spray starch	0.1	0.4	Ma.
		IML contaminated with Krylon			
-011	0.46	spray starch	0.0	1.3	
-012		IML contaminated with Krylon	HB	HB	N/A

Table 11- MISC-794-484 Test Results

		spray starch			
		IML contaminated with MS-143			
-013	0.36	Mold Release Agent	0.2	0.4	
		IML contaminated with MS-143			
-014	0.4	Mold Release Agent	0.4	0.5	P
		IML contaminated with MS-143			Iren
-015	0.33	Mold Release Agent	0.5	10.0	
		IML contaminated with MS-143			N/A
-016		Mold Release Agent	НВ	HB	
		IML contaminated with MS-143			N/A
-017		Mold Release Agent	НВ	HB	
HB 1	Indicates te	st completed at HB a	and no	data was	available

APPENDIX C: SIP ADHESION TO THE IML OF THE TILE

Cohesive bond failure mode:



This is the expected result when the SIP and IML are debonded. It reflects the optimal strength.

Adhesive bond failure mode:



The subnominal bond anomaly addressed in this paper is an occurrence of an adhesive failure. This failure is identified by reduced peel strength, unknown tensile properties, and low $\left(<\frac{4lb}{in}\right)$ peel strength.

Mixed bond failure mode:



This is a combination of the adhesive and cohesive failure modes. There is some densification damage on SIP removal. The mixed failure mode is distinguished from the adhesive failure mode as it has less than 50% adhesive failure.

APPENDIX D: HISTORICAL DOCUMENT REVIEW – ELIMINATION OF POSSIBLE SUBNOMINAL BOND CAUSES

• Workmanship (Densification Process)– Not Eliminated

- Issue: The technician densifying the tile may have made an error resulting in the introduction of a contaminant, or insufficient densification.
- Consequence: Contamination on IML surface may inhibit tile to SIP bond. Insufficient densification may lead to improper SIP to tile adhesion.
- Eliminated: Maskant adhesive (which masks the sidewalls of the tile to prevent the densification slurry from contaminated the sidewall) is a tape, either Mystic 7000, 7001, CHR G 565 or 3M #361, and it may have been contaminated. Additionally, if the maskant adhesive was allowed to sit on the tile surface too long, excessive build-up of the tape bond-strength to the tile coating would occur and the tile could become damaged upon maskant removal. Contamination in the working container was also not a cause as both proper and improper SIP/IML adhesion came from containers from the same lots. Additionally, both adhesive and cohesive bond failures were produced by technicians sharing a common work area. Finally, the document review found that weight pickup was within specification requirements for tiles exhibiting both types of bond failure.
- Not eliminated: It is possible that the technicians used contaminated brushes or the SIP excess slurry wipe contaminated the tile.

• Material Anomalies (Densification Process) – Not Eliminated

- **Issue**: An anomalous material may have been used in the densification process.
- Consequence: An anomalous material may lead to inadequate densification of the tile IML, resulting in an adhesive bond failure.
- Eliminated: Alcohol used by technicians was carried in non-leaching plastic bottles and alcohol from the same lot was used on tiles that experienced both types of bonds, thus plasticizers in the alcohol can be eliminated. The MB0115-011 Ludox used was, according to the document review, maintained at proper temperature levels and the vendor was found to meet all specification requirements. The document review revealed that the MB0115-036 Silica powder vendor and the MB0115-022 Tetraboride powder met all specification

requirements. Furthermore, the document review found no evidence of expired shelf-life for any material.

Not Eliminated: Material anomalies in the densification slurry, the MC0115-036 Silica Powder, the MB0015-036 Silica Powder High Iron/Crystalinity and the Alcohol were not eliminated as possible subnominal bond factors in the document review.

• Process Deficiencies (Densification Process) – Not Eliminated

- > **Issue**: There may be a deficiency in the densification process.
- **Consequence**: Tile densification may be inadequate.
- Eliminated: The weight pickup requirement is adequate as proven by the ratio of cohesive to adhesive bond failures since some cohesive bond failure tiles had lower weight pick-up than adhesive bond failure tiles. SIP excess slurry wipe process was also acceptable because scrap SIP was used as a wiper and laboratory tests on that scrap did not correlate material extracted from SIP with the bond surface.
- Not Eliminated: The brush cleaning requirement is still a possible process deficiency, as a contaminated brush could lead to adhesive failure.

• Workmanship (Factory Waterproofing Process) – Not Eliminated

- Issue: The technician working the waterproofing process may have made an error resulting in insufficient waterproofing.
- Consequence: Waterproofing improves RTV adhesion to tile IML. An error in the waterproofing process may lead to an adhesive bond failure.
- Eliminated: The pickup weight for each run was within specification limits and a water drop test was done on each tile, thus the pickup weight is not a factor. Additionally, using the wrong cloth can be eliminated as a fiberglass cloth was always used.
- Not Eliminated: The tile may have become contaminated during the waterproofing process.

• Material Anomalies (Factory Waterproofing Process) – Not Eliminated

- > **Issue**: An anomalous material may have been used in the waterproofing process.
- Consequence: An anomalous material may lead to inadequate waterproofing, resulting in improper RTV adhesion to the tile IML.
- Eliminated: The fiberglass cloth could not have been contaminated because specification requires the cloth be discarded after 3-5 runs. The Silane had not exceeded its self-life, as it had been tested per the requirements of MB0115-020, 12 months minimum from shipment from vendor. Additionally, the Silane was stored properly in non-leaching plastic bottles, with all runs utilizing the same procedure, and no discrepancies were noted upon receiving and inspecting the Silane received from the vendor. The acetic acid was bought commercially and all runs utilized the same procedure, therefore material anomaly in the acetic acid is highly unlikely.
- Not Eliminated: The purity of the Silane used is unknown thus leaving open the possibility of contamination.

• Oven – Not Eliminated

- ▶ **Issue**: An issue with the waterproofing oven(s) may have gone undetected.
- Consequence: An anomalous waterproofing oven may result in inadequate waterproofing, leading to improper RTV adhesion to tile IML.
- Eliminated: Temperature variability within the oven was not a problem as the ovens were calibrated by performing a five point temperature profile. Chemical dispersion within the oven was also no factor in the subnominal bond issue as all waterproofing runs had weight pickup values that met specification requirements and each tile passed a water drop test. The ovens were also cleaned and maintained properly, undergoing thorough cleaning every four runs. Any oven anomalies would have resulted in a vacuum discrepancy and/or failure to meet a weight pick-up requirement. Cohesive and adhesive bond failures both occurred

from tiles that had undergone the same waterproofing runs, so oven temperature extremes was not a factor.

Not Eliminated: The oven vacuum pump could have malfunctioned, and a small malfunction could have gone undetected, thus leading to inadequate waterproofing which could inhibit RTV adhesion to the tile.

• Workmanship (SIP Bond Process) – Not Eliminated

- ▶ **Issue**: The technician may have made an error while bonding SIP to the tile.
- Consequence: An error during the SIP bond process may lead to an adhesive SIP to tile bond failure.
- Eliminated: Too much RTV was not applied during the bond process since no DRs were generated; additionally, too much RTV would not lead to an adhesive failure. The same is true for too much pressure application. The correct catalyst was also used since the wrong catalyst would be detected by rapid cure prior to application, or failure to achieve Shore A hardness. Finally, if SIP slipped prior to cure, SIP was removed and new SIP was installed without heat cleaning tile. If SIP slip was discovered after cure, SIP was removed, tile heat cleaned, and new SIP installed. Proper and improper bonds were intermingled on bond tables.
- Not Eliminated: The RTV may have been incorrectly applied, the pressure application may have been done incorrectly, and the quantity of the catalyst used may have been incorrect.

• Contamination (SIP Bond Process)– Not Eliminated

- **Issue**: Contaminated materials/tools may have been used in the SIP bond process.
- **Consequence**: Contamination may inhibit RTV adhesion to the tile IML.
- Eliminated: The RTV was not unusually contaminated as disclosed in laboratory resting of removed RTV. Additionally, the tools used in the SIP bond process were not a factor as the SIP bond room is a controlled environment and cleanliness rules are in effect. Contamination is highly unlikely.
- > Not Eliminated: It remains a possibility that the IML was contaminated

• Environmental Conditions (SIP Bond Process) – Not Eliminated

- > Issue: Did temperature/humidity/airborne particulate affect SIP to tile bond?
- Consequence: Low temperature/humidity can retard the RTV cure, resulting in pressure being removed prior to full cure. High temperature/humidity will accelerate the RTV cure, possibly causing RTV surface to skim over prior to pressure application. High temperature/low humidity may cause thin film adhesive to lose moisture during bond process. Airborne particulate deposited on the tile IML may inhibit adequate RTV adhesion to the tile.
- > Not Eliminated

• Vehicle Location – Not Eliminated

- > Issue: Are adhesive bond failures dependent on location of vehicle?
- Consequence: A specific location/environment may lead to an adhesive bond failure.
- Eliminated: Orbital flight and ferry flight could not have caused this anomaly as there is no mechanism for bond deterioration that could affect only the IML to SIP bond under these conditions. The flight processing facility is also not an issue for the same reason.
- Not Eliminated: All failure bonds were Palmdale bonds, though many more proper bonds were documented in Palmdale (28 improper/142 proper).

• Age Issues – Not Eliminated

- Issue: Has time degraded the SIP to tile bond through RTV reversion or by a reaction between DMES and RTV?
- Consequence: RTV reversion or degradation will lead to an adhesive bond failure.
- Eliminated: The OMRS vehicle sampling has not identified age related degradation. Laboratory tests have not identified RTV reversion or other types of degradation.

Not Eliminated: It is still possible that the SIP to tile interface degrades over time, though not likely.

• Production Units (Tile Fabrication Process)- Eliminated

- Issue: Adhesive bond failures may be dependent on production unit utilized to machine tile.
- Consequence: All tile manufactured from discrepant production units would exhibit an adhesive failure bond.
- Eliminated: Density gradients, PU contamination and the PU being under sintered were eliminated as possible causes because a single PU produced tiles that were both cohesive and adhesive. Additionally, heat clean would remove contaminants. Pus all met specification requirements, including those for cleanliness composition. The tile "coat and fire" process would have resulted in failure had the PU been contaminated.

• Initial Tile Fabrication - Eliminated

- Issue: Initial steps in the tile fabrication sequence may have led to adhesive bond failures.
- Consequence: Tile manufactured using discrepant material/machine may exhibit an adhesive bond failure.
- Eliminated: Waterproofing was eliminated as a cause because discrepancies which occurred during the 1st run would result in inadequate densification slurry penetration and the tile would not progress. Coating/firing was eliminated as firing at 2200°F would either remove the contaminant or cause a coating anomaly. A difference between IML machining NC and tracer pattern based tile machining was eliminated since bottled GN2 was used for both, and both produced both adhesive and cohesive SIP to tile IML bond failures. Finally fabrication location (LMSC vs. PLMD vs. KSC) was eliminated since both LMSC and PLMD fabricated tiles exhibited adhesive bond failures. No failures were noted on tile fabricated at KSC.

• Equipment (Densification Process) – Eliminated

- **Issue**: Faulty equipment may have been used in the densification process.
- Consequence: An adhesive SIP to tile bond failure may have been caused by an equipment error.
- Eliminated: Faulty equipment was eliminated as a possible cause because it is very unlikely that a faulty oven could contribute to an adhesive bond failure. Additionally, IML dusting utilized GN2, therefore contaminates are unlikely to be deposited on the IML.

• Process Deficiencies – Eliminated

- **Issue**: There may be a deficiency in the waterproofing process.
- Consequence: Waterproofing may be inadequate to ensure proper RTV adhesion to tile IML.
- Eliminated: The process is stable based on the ratio of cohesive to adhesive bond failures.

• SIP Lot Number – Eliminated

- Issue: Contaminated or anomalous transfer coated SIP may have been bonded to tile.
- Consequence: Contamination, or a discrepant transfer coat, may lead to an adhesive SIP to tile bond failure.
- Eliminated: Adhesive bond failures were not confined to a given SIP lot. Furthermore, failures were between the IML and the RTV, not the transfer coat and the RTV.
- Fault Tree Process Deficiency Eliminated
 - Issue: A combination of factors, all within specification requirements, could have combined to produce adhesive bond failures.

- Consequence: Adhesive SIP to IML bond failures could have been produced while staying within process specification requirements.
- Eliminated: The analysis of the ratio of proper to improper bonds proves the process is stable.

• Storage/Transfer – Eliminated

- **Issue**: The tile IML may have been contaminated during storage transfer.
- Consequence: Contamination may lead to an adhesive SIP to tile IML bond failure.
- Eliminated: Laboratory testing did not show unusual amounts of contaminant on the tile IML, SIP, or RTV indicating that there was no contamination during storage or transfer.

• Prefits – Eliminated

- ▶ **Issue**: The tile IML may have been contaminated during first and/or second prefit.
- **Consequence**: Contamination may lead to an adhesive SIP to tile bond failure.
- Eliminated: Wax, mosite, plastic wrap, and hydraulic fluid could not have been causes of the subnominal bond problem as wax and mosite were not used in PLMD during the OV-105 build, plastic wrap was not used between the 2nd prefit and the SIP bond and the tiles were installed prior to the presence of hydraulic fluid. The SIP could not have been contaminated at that time because in house processes for contamination were in effect and visibly contaminated SIP would have been discarded. Finally the environment in the bay was not controlled, but laboratory analyses did not detect contamination on the IML, SIP, or RTV at that time.

• Ship from Vendor (LMSC) – Eliminated

- **Issue**: The tile IML may have been contaminated prior to shipment to PLMD.
- **Consequence**: Contamination may lead to an adhesive SIP to tile bond failure.

Eliminated: All the tiles were densified and SIP'd at PLMD, thus eliminating shipment as a possible source of adhesive failure.

• FC723 Waterproofing. – Eliminated

- Issue: The tile IML may have been contaminated with brush- on waterproofing compound (FC723) prior to SIP bond.
- **Consequence**: Presence of FC723 on the tile IML would inhibit SIP bond.
- Eliminated: The FC723 brush on waterproofing could not have caused the subnominal bond as PLMD did not normally repair tiles after waterproofing and all adhesive bond failure tiles were free of inserts and IML repairs.

• Location on Vehicle. – Eliminated

- **Issue**: Contamination of the tile IML may be dependent on area of vehicle.
- **Consequence**: Contamination may lead to an adhesive SIP to tile bond failure.
- Eliminated: Adhesive bond failures have been found in multiple locations on the vehicle.

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