

Spacecraft Adhesive Residue On-Composite Analysis

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Define all symbols used in the abstract. Do not cite references in the abstract.

Nomenclature

ASTM = American Society for Testing and Materials

AWD = Analytical Working Distance

IOZ = *Industrial Operations Zone in the O&C Highbay*

IPA = Isopropyl alcohol (isopropanol)

KSC = Kennedy Space Center MEK = Methyl ethyl ketone

O&C = Neil Armstrong Operations and Checkout Building

PLM = Polarized light microscopy SEM = Scanning electron microscopy

Abstract

Tapes are used on space vehicles for numerous reasons; closing ports, labeling, and even temporarily attaching ground servicing equipment to the spacecraft. This project stemmed from the need to determine which tapes are most effective for our customer's space vehicle during its ground processing stage. During ground processing, there are multiple stages of processing where workers need to use tape to temporarily stabilize or close-off components for a matter of seconds, days, weeks, or even a few months. After peeling these tapes off the flight hardware, the residue left behind by the tape poses potential risks that can lead to incidents, which may not be easily noticeable to the engineers. It is important to identify these issues prior to space flight. The purpose of this project is to assist in research to create an accessible reference for which tapes are preferred on common flight hardware surfaces. Five different substrates and eight tapes were selected for evaluation. The selected substrates were chosen because they comprise the most surface area on the vehicle. The tapes selected were either heritage or new contenders. The adhesive studies were performed for multiple dwell times to study the tendency of different tapes to leave residue behind on various substrates and quantifying that residue. Additional analytical tests were performed to supplement the main objective of the project. These tests included adhesive solubility, peel adhesion, and scanning electron microscopy (SEM) analyses.

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I. Introduction

A. Purpose

When sending components into space, it is important to consider everything, big or small. When a job as time consuming and labor intensive as constructing a spacecraft, it is essential to be as careful and aware of potential hazards and contamination. During the ground processing phase of a space vehicle's journey, adhesive tapes are applied to its surfaces to hold plastic films in place, temporarily close ports/holes, and secure foams in place to prevent contamination. However, the residues these adhesive tapes leave behind can cause contamination issues. If these residues are overlooked, and make it past ground processing into space, the residue has the possibility to off-gas and compromise the ability of the hardware to perform efficiently or at all. Damages off-gassing does to the hardware include settling on optics and affecting thermal control systems. This can also risk the health and safety of the brave astronauts sent into space. These threats also have the potential to compromise the spacecraft's processing schedule by causing a work stoppage, which could ultimately lead to a launch delay. Currently, the customer has no data on adhesive residue transfer or any method on how to quantify it. Lacking this information leads to misusage of tapes, and ultimately results in a time-intensive and costly cleanup. Therefore, this project is integral to the safety and efficiency of building, protecting, and launching spacecraft, as well as ensuring the well-being of astronauts who ride in them. The purpose is to use this research to develop an appendix of tapes that are safe to use on specified flight hardware surfaces. This will prevent contamination issues by creating an easy and accessible reference for engineers to refer to quickly. After completion, this reference will help reduce the amount of tape contamination issues and establish a resource for years to come.

B. Scope

In this particular case, the customer needed to identify the tapes they should use on their spacecraft in order to prevent incidents of contamination from occurring later on. The study is comprised of 8 different tapes that all vary in color, strength, backing material, and adhesive material. Some of these tapes were chosen based on their low tact to reduce residue deposits and to compare to tapes with higher tact. The five substrates included in this study are representative of actual spacecraft surfaces. These eight tapes are standard-pressure adhesives. After preparing the tape/substrate specimens, they were placed in the Industrial Operations Zone (IOZ) for a set time period. The IOZ is where the spacecraft is being built, so it is best for the tapes to be tested in the exact environment for its field of use. Experiments performed in this study include adhesive/solvent solubility, SEM analysis, and peel adhesion tests.

II. Methods

For this project, eight tapes and five substrates were evaluated (see Tables 1 and 2). The tapes are all standard pressure-sensitive adhesives (see Figure 1). The tapes are 1" wide and the coupons (substrates) are 2" x 2". The adhesive tape and substrate combinations were tested over time intervals of 1 week, 1 month, 4 months, and 6 months. Each tape and substrate combination was tested a redundancy of three times for each time period. These tapes were tested under the conditions and time intervals based on actual field use.



Figure 1. Tapes (from left to right): 3M 471, 3M 481, FB-1R, KPT-1, 3M 5490, 3M 851, CHR 734, BA 12989

Table 1. Substrates

Titanium	Koropon on Aluminum	Elimstat on Vamac	Composite (carbon epoxy face sheet with copper mesh)	Composite (carbon epoxy face sheet) rough side

Table 2. Tape Names with Backing and Adhesive Materials

	Tapes								
	3M 471	3M 481	3M 851	3M 5490	BA 12989	CHR M734	Flashbreaker- 1R	KPT-1	
-								4 4 4 4	
Backing	vinyl	polyethylene	polyester	polytetrafluoroethylene	polyester	polyester	polyester	polyimide	
Material									
Adhesive	rubber	rubber	rubber/silicone	silicone	acrylic	rubber	rubber	silicone	
Material									

A. Preparation Techniques

1. Optical Microscopy

Preliminary preparation techniques are required for efficient use of the scanning electron microscope. Optical microscopy is the best start to being comfortable with any microscope. Optical microscopy, often referred to as light microscopy, uses visible light and a variety of lenses to magnify very small objects that are not observable to the naked eye. For example, the macroscopic characteristics of sand are observable to the naked eye, but surface detail and morphology are not. By using optical microscopy, the surface details and morphology of sand particles can be observed and studied. Optical microscopy has 3 standard settings of light: oblique, transmitted, and coaxial

illumination. Each light setting is different in terms of the way is broadcasted. Oblique lighting is cast upon a subject creating a high contrast. Transmitted lighting is light projected from the bottom of the microscope. The light passes through a condenser and the specimen, creating a high illumination. Coaxial illumination where a beam of light is guided through the optics and reflected from a sample, works best for smooth and reflective samples. It is especially useful to observe fine cracks or surface quality if needed. Coaxial illumination is also the only technique applicable to observe delaminated glass. Another important procedural technique in optical microscopy is determining your analytical working distance (AWD) and depth of field. The analytical working distance is the distance between the

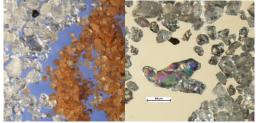


Figure 2: Sand vs. Soil with transmitted lighting and sand particles under oblique lighting with big particle with high birefringence. Both images were taken with the Nikon SMZ1500 microscope.

lenses of the microscope and the specimen when the specimen is clear and in focus. Depth of field is the region actually in focus determined by magnification and numerical apertures. A numerical aperture is a measure of a microscope's ability to gather light and observe fine specimen detail at a fixed object distance. Optical microscopy is also best for single particle handling because of the view, space, and clarity achievable with a stereo microscope. Learning the different lighting techniques, determining your AWD and depth of field, and understanding the tools of a stereo microscope are all techniques best learned on an optical microscope before moving on to more advanced microscope applications.

2. Polarized Light Microscopy

Polarized light microscopy (PLM) is a contrast-enhancing microscopy technique. It is specially designed to observe specimens that are visible primarily due to their optically anisotropic (crystalline) character. When a

sample is anisotropic, polarized light can be used to determine characteristics such as degree of birefringence, refractive index, and sign of elongation. The prevailing technique for sample illumination for polarized light analyses is Köhler illumination. It provides optimum contrast and resolution and is a defining characteristic of polarized light microscopy. This type of illumination is achieved by focusing and centering the light

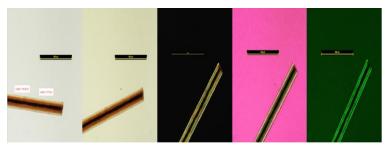


Figure 3. Monkey fiber under Polarized light (left to right: no polar, single polar, cross polar, cross polar with red wave plate, Becke line with green filter)

path and spreading it evenly over the field of view, which requires opening and sharpening aperture.

B. Tape Application and Peel Adhesion Test

1. Tape Application

The American Society for Testing and Materials (ASTM) D3330 "Standard Test Method for Peel Adhesion of Pressure-Sensitive Tape" Method F was used for tape application and removal. Test Method F gives a measure of adherence, when peeled at 90° angles, to a standard steel panel or other surface of interest. Prior to each tape application, the Koropon on aluminum, composite, and titanium substrates were cleaned with acetone. Isopropyl alcohol (IPA) was used to clean the Elimstat on Vamac substrate since acetone removed the Elimstat coating, and was therefore incompatible. The tapes were applied to the coupon by hand and a tab was formed at the end for the

future peel adhesion tests. As stated previously, all of the tape and coupon samples were stored in the IOZ since this is where the spacecraft is assembled and where the tapes will be used. After the designated time period, the samples were collected and the tapes removed at a rate of 20 in/min using a Mechanical Instron 5900R Model 4507 testing load frame positioned 90° to the coupon.



Figure 4. Coupons (left to right: Koropon, composite-rough, Elimstat on Vamac, and titanium) after several tests that are slightly damaged/discolored by cleaning.

2. Peel Adhesion Test

After the designated test exposure times, the samples were retrieved from the IOZ, and the tapes were immediately removed to measure peel adhesion using the Mechanical Instron 5900R, Model 4507. The 4507 Instron is designed to evaluate the mechanical properties of materials and components. The Instron measures the adhesion strength of the tape as it is peeled from the substrate at 90°. When peeling the tape, it collects the pound by force over

width of the tape (lbf/in) for each test. As stated previously, each tape and substrate test were repeated for a total of three times. It takes about seven minutes to complete test, including inserting and removing the tape and substrate.



Figure 5: Tapes/coupons samples in mechanical testing room after IOZ departure (left). The 4507 Instron before peel test with sample already prepped (middle and right).

C. Adhesive Solubility Test

The adhesive solubility tests were performed using (weakest to strongest in terms of residue removal) isopropyl alcohol (IPA), acetone, amyl acetate, and methyl ethyl ketone (MEK). This is needed to determine what solvent should be used to remove a particular adhesive. Using a syringe needle, 1 drop (approximately 10 microliters) of solvent was placed on the adhesive. After 10-15 seconds of solvent exposure, a needle was used to score an "X" onto the surface to observe how much the adhesive had dissolved. As a control, an "X" was scored onto the adhesive in the absence of solvent as a reference for comparison to adhesive solubility test end results

D. Scanning Electron Microscopy Analysis

After peel adhesion tests were completed, the residue left on the coupons was examined by scanning electron microscopy with energy dispersive spectroscopy (SEM/EDS). The two detectors used for the SEM analyses were the backscatter (BSD) and secondary (SE) electron detectors. BSD is used to visualize elemental contrast and collect elemental spectra. The SE detector is used to image the topography of the sample. Certain adhesive and substrate combinations are more easily identified topographically than by elemental contrast, so both detectors were utilized in the SEM analysis.

III. Results

A. Peel Adhesion Results

The Mechanical Instron software generates the average peel adhesion (lbf/in) for each test. After collecting the data from each tape pull, the integration boundaries were redefined to exclude the gradual start and end periods. The integration over these new boundaries provided the final peel adhesion value. This value was averaged between the three redundant tape/substrate specimens for each time period (1 week, 1 month, 4 months, and 6 months). Table 3 contains the collected results over time of each tape and the substrates; these tables are categorized by substrate type and the time interval is recorded by days. If a tape pull peels or disturbs the substrate surface in any way, the tape/substrate compatibility is an automatic failure. This also means SEM analysis is no longer needed and the tape can be deemed prohibited or minacious to the substrate. It lets the customer know that this tape should not be used on a hardware with that particular material.



Figure 6. Bron and Elimstat on Vamac automatic failure example

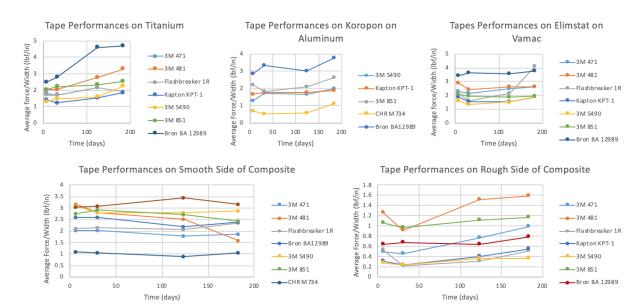


Table 3: Combined Table of All Tape/Substrate Peel Adhesion Test Results

Based on the overall performance of all the tapes on each substrate, it is clear that the average force over the time periods is not trending in one direction for all the tape and substrate combinations. It did not take much effort for the Instron to peel these off the coupons, especially on the rough side of the composite. The roughness and irregularity in the substrate's texture makes it very hard for the tape to really cling to the surface. This is why the average force for all tapes is much lower for the rough side than on the other substrates. However, on average, when the tapes were attached for a longer amount of time, it took more force to pull off the coupons, including the rough side of the composite. By logic, we expected these results because the adhesive would have more time to settle on the surface, making it harder to detach.

B. Adhesive Solubility Test Results

The results of the adhesive solubility test showed that acetone, amyl acetate, and MEK were much more successful at dissolving the adhesives than IPA. All of the adhesives were relatively insoluble in IPA. This is to be expected since IPA is a polar solvent and the adhesives are relatively nonpolar. Findings like these are important since the primary solvent used to remove adhesive residues in the IOZ is IPA. The rubber adhesives (3M 471, 3M 481, CHR M734, and FB-1R) were most soluble in amyl acetate and MEK. The pure silicone adhesives (3M 5490 and KPT-1) were most soluble in acetone and MEK. Interestingly, 3M 851, the rubber and silicone hybrid adhesive was most soluble in amyl acetate and MEK. Bron BA 12989, the acrylic adhesive was only slightly soluble in acetone and amyl acetate.

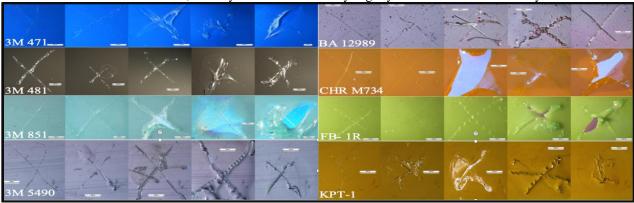


Figure 7. Adhesive Solubility Test Results (left to right: Original, IPA, Acetone, Amyl Acetate, and MEK)

C. Scanning Electron Microscopy Analysis

Due to time constraints and SEM malfunctions, only 3M 481 on titanium samples have been analyzed. Each coupon takes about 15 minutes to be examine and gather findings. In Figure 8, the images on the left show 3M 481 on titanium after 1-week dwell time and the images on the right shows them after 6 months. A huge difference can be seen in the amount of residue remaining after the tape pull. As expected, the longer the tape is adhered, the more residue it will leave behind once removed. However, on the 6-month sample, around the residue appears to be an oil associated with it. This oil is most likely originating from the adhesive material. Findings like these are crucial to characterizing different adhesives' performances over time.

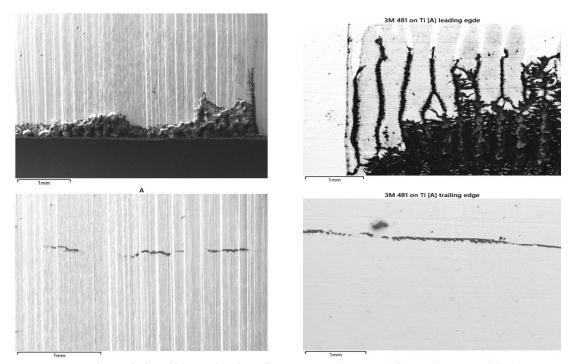


Figure 8. 3M 481 on Titanium SEM Images 1 week (left) vs. 6 months (right)

IV. Future Work

In the future, SEM/EDS analyses will need to be completed for the remaining samples. In addition, there will need to be more research and effort to solidify a method for quantifying adhesive residue. Also, there is currently a preliminary rating scale that works well, but there is still a process and more trials to be done to actually standardize and perfect the rating criteria and reliability for public export. When it came to the adhesives' solubility, amyl acetate was effective in dissolving most of them. Unfortunately, it is currently not allowed for use in the IOZ and will be looked into for future use.

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