# The Analysis of Adhesive Residue Failure

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# The Analysis of Adhesive Residue Failure

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**Adhesives have always been, and will continue to be, a crucial component of flight hardware processing. Unfortunately, adhesives have a long standing history of being a source of costly contaminations. There are currently no existing documents that can aid a technician in discerning the proper adhesive/substrate combinations, nor the optimal solvent to use when removing adhesive residue. This project seeks to evaluate the performance of adhesive tapes on various flight hardware substrates in order to create an easy-to-use guide for spacecraft ground processing. The analyses performed were the adhesive/solvent solubility, scanning electron microscopy (SEM) examinations, and peel adhesion tests. The solubility tests showed that amyl acetate, acetone, and methyl ethyl ketone (MEK) were the most successful in dissolving the adhesives. The SEM data gives preliminary insight into the morphology of the residues. Lastly, the peel adhesion testing proved that there is no relationship between the length of time the adhesive was applied and its adhesive strength.**

## Nomenclature

*OM = Optical Microscope*

*IPA = Isopropyl Alcohol*

*MEK = Methyl Ethyl Ketone*

*SEM = Scanning Electron Microscope*

*BSD = Backscattered Electrons*

*SE = Secondary Electrons*

*µm = micrometers or $10^{-6}$ meters*

*cm = centimeters or $10^{-2}$ meters*

*mm = millimeters or $10^{-3}$ meters*

## I. Introduction

Adhesives have been crucial to spaceflight programs from the beginning and continue to be used in a plethora of settings. Unfortunately, with adhesives comes the residues they leave behind which can be due to a wide variety of factors. Variables such as the environment, plasticizers, surface energy of the substrate, and adhesive strength can all influence the amount of residue left behind. It is a well-known fact that spacecraft need to be free of contaminants in order to reach optimal performance, however, there is currently no document in existence that describes the probability of a given tape to leave a residue. Additionally, there is no existing document to aid in the proper removal of any residue left behind. This project aims to collect data for use in an upcoming adhesive-residue-transfer data section in the Kennedy Technical Instruction 5212 "Material Selection List for Plastic Films, Foams, and Adhesive Tapes". The addition of this section will act as a guide for pairing adhesives with the proper flight hardware surfaces (or substrates)

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and reduce the number of adhesive contamination incidents. Originally, this project was born when the Orion Program sought to identify the prime tapes to use on their vehicle. The study looked at eight tapes and five substrates, all of which were chosen by Lockheed Martin Orion Contamination Control Engineering. The test substrates in this study were chosen since they comprised the majority of the surface area of the Orion vehicle. All of the tapes listed in this paper are standard pressure-sensitive adhesives. This experiment is comprised of several studies, but this paper will only focus on adhesive/solvent compatibility and peel adhesion tests.

## II. Methodology

### A. Optical Microscopy

The two primary functions of an optical microscope is to create magnified images and to illuminate samples. To create a pristine magnified image, there are several steps to be taken. This includes: getting the image to be clear and sharp, adjusting the magnification, and bringing the specimen into focus. To properly illuminate the sample, there are basic functions, such as, supplying light, collecting light, and changing the light intensity. Collectively, all the mentioned functions are referred to as an "illumination optical system". Through this system a projected image is obtained and can be viewed through the lens, with the naked eye, or on a computer.[2] For this particular project, the microscope was used primarily for the solubility testing performed on the adhesives of the various tapes. No sample preparations were required. The solvents tested were: amyl acetate, acetone, IPA, and MEK. This test was performed by applying a drop of solvent onto the adhesive and after fifteen seconds, drawing an X into the adhesive. Under the OM the degree of solubility was determined by how much the adhesive was visibly removed/dissolved and then photo documented. These pictures were then compared to an unaltered piece of adhesive.

### B. Scanning Electron Microscopy

The scanning electron microscope utilizes a focused electron beam to produce signals and high resolution images from the surfaces of solid samples. These signals give information about the external morphology and chemical composition. Typically data is collected from selected areas of a specimen. This study made use of the signals produced from secondary electrons and backscattered electrons. Secondary electrons are useful for showing the morphology and topography of a sample. Backscattered electrons are useful for illustrating the contrast in the composition of a multiphase sample.[3] For this project, the SEM was instrumental in capturing adhesive residue that was too miniscule to be viewed on the optical microscope. After the tape pulls were conducted the coupons were placed directly into the vacuum chamber of the SEM and analyzed under high vacuum. The coupons were scanned and the adhesives were rated on how much residue they left behind. Additionally, if the coating of the coupon was removed during the tape pull process it was rated an automatic failure and was not looked at under SEM.

### C. Instron Mechanical Testing

Instron manufactures numerous electromechanical instruments that perform tests such as tensile, compression, peel, tear, and bend. These instruments have also been designed to perform industry standardized tests on differing materials. Their product line offers a range of sizes and force capacities to accommodate testing on all force systems. In particular, this study utilized the ASTM D 3330 Standard Test Method for Peel Adhesion of Pressure-Sensitive Tapes, Test Method F – Single Coated Tapes at 90° Angle. To prepare the substrates, the titanium, Koropon on aluminum, and composite coupons were cleaned with acetone. The Elimstat on Vamac coupons were cleaned with IPA. Six inch strips of each tape were cut and applied to the coupons, using half of the strip to fold on itself, creating a tab. The coupons were then left to sit for differing time spans of one week, one month, four months, and six months. After the allotted time has passed, the coupons are placed into a load frame and clamped down for the adhesion strength test. Making sure the strip of tape is parallel with the moving jaw of the load frame, the tab on the coupon is placed into the jaw at a 90° angle. The pull is then performed at a rate of 20 in/min. [4]

## III. Results and Discussion

### A. Adhesive Solubility Testing

These tests were performed to provide complimentary solubility data to the appendix. It is important for technicians, who are working hands-on with the hardware, to know what solvent to use to remove a particular adhesive. In this test, 1 drop of solvent was applied directly to the adhesive, allowed to sit for fifteen seconds, and had a needle score an X into the area to determine how much the solvent dissolved the adhesive. All images were obtained on the optical microscope.

#### *1. Bron BA 12989*

It can be seen below that the IPA did not dissolve any of the adhesive. The X made looks nearly identical to that of the adhesive containing no solvent. The amyl acetate, acetone, and MEK appear to be slightly dissolving the adhesive. While a deeper X was able to made, the remainder of the adhesive was left intact. Bron BA 12989, which is an acrylic adhesive, was not completely soluble in any of the solvents, however, it was slightly soluble in amyl acetate and acetone.

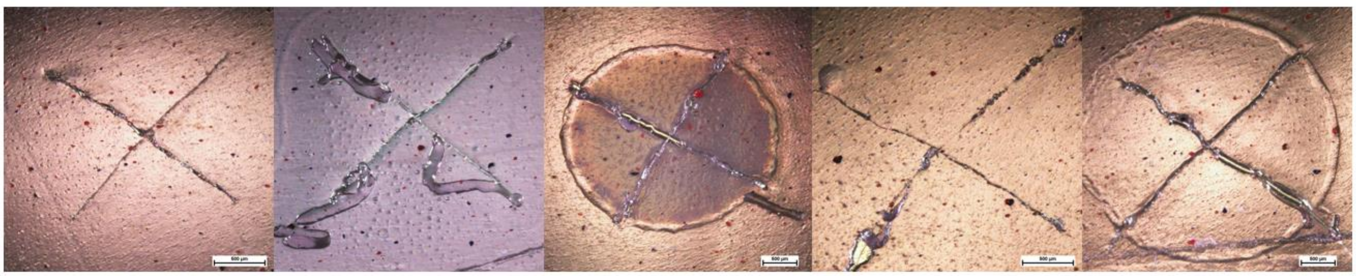


**Figure 1:** (From left to right) No solvent, amyl acetate, acetone IPA and MEK.

#### *2. 3M 471*

IPA was not effective on this adhesive. Again, there is no noticeable difference between the IPA X and the original image. The amyl acetate partially dissolved the adhesive, leaving a pile of sticky residue where the drop was applied. Both the acetone and the MEK strongly dissolved the adhesive. Not only was a ring created from where the solvents were applied, but the X's are deep. The strung out appearance of the X's suggests that the solvent was able to penetrate well into the adhesive. 3M 471, a rubber adhesive, was soluble in amyl acetate, acetone, and MEK.

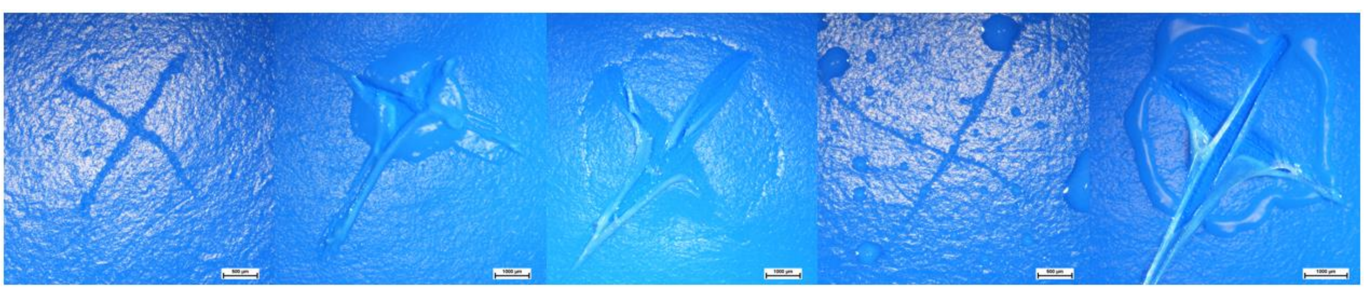


**Figure 2:** (From left to right) No solvent, amyl acetate, acetone IPA and MEK.

#### *3. FlashBreaker-1R*

IPA and Acetone were the least effective solvents. The X's drawn are very faint and not deep. The rubber adhesive of FB-1 was soluble in amyl acetate and MEK. Both solvents showed a high level of solubility which can be seen by the pile up of adhesive formed from drawing the X on the tape. In the amyl acetate image, no discernable X can be seen, meaning it dissolved the adhesive the best.

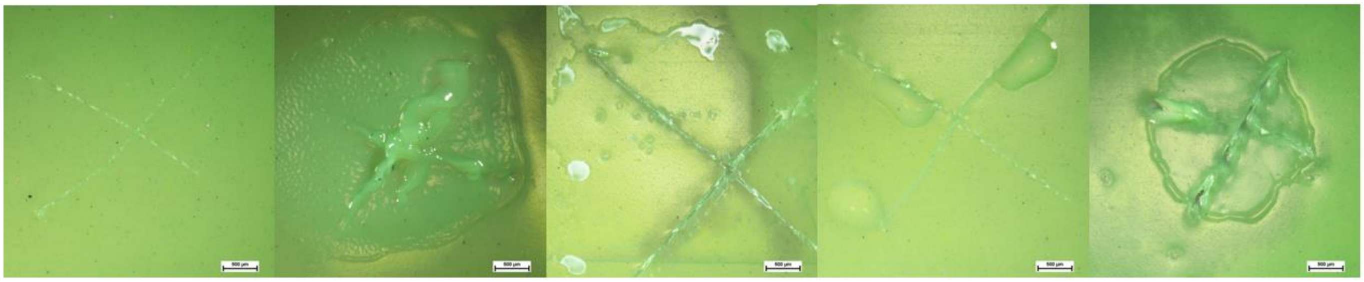


**Figure 3:** (From left to right) No solvent, amyl acetate, acetone IPA and MEK.

*4. 3M 5490*

3M 5490's adhesive material is pure silicone. It was determined the IPA did not dissolve any of the adhesive. It was discovered that the silicone was most soluble in acetone and MEK. Residual strips of adhesive can be seen dangling where the X was made but the majority of the adhesive was left intact. The acetone seems to have dissolved the adhesive greatly, allowing for piles of residue to be formed around the X area. Acetone would be optimal for removing 3M 5490 adhesive residue.

**Figure 4:** (From left to right) No solvent, amyl acetate, acetone IPA and MEK..

*5. Kapton*

KPT, another silicone adhesive, was also insoluble in IPA. MEK performed marginally better, creating minimal build-up of adhesive where the X was drawn. AA appears to have had a reaction with the adhesive rather than dissolving it making this result unusual. Acetone dissolved the adhesive the best allowing for clear separation of the adhesive from the tape backing. Overall, the silicone adhesive was most soluble in acetone and MEK.

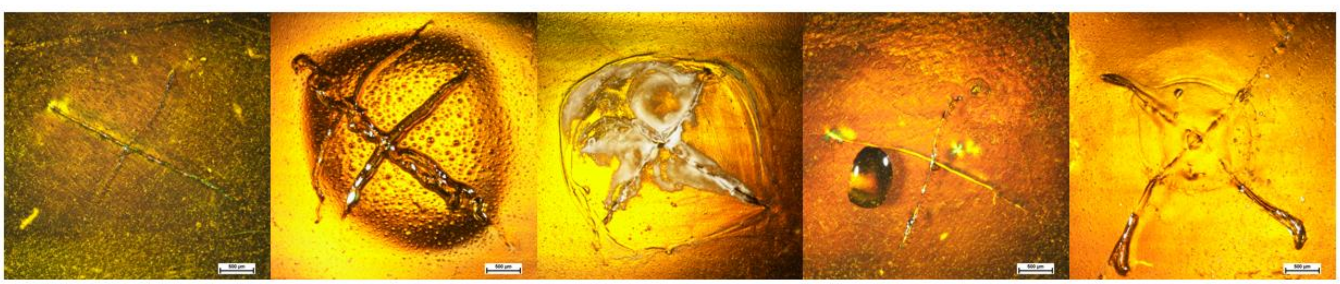


**Figure 5:** (From left to right) No solvent, amyl acetate, acetone IPA and MEK.

*6. 3M 851*

3M 851's adhesive material is a rubber/silicone blend. IPA did not dissolve any adhesive. It was slightly soluble in acetone and could still be used to remove small amounts of residue. This solvent did not cause the residue to become fully fluid, as seen in the images of the MEK and amyl acetate. With both solvents, the residue was dragged instead of remaining stiff and allowing a clear X to be seen. Both contain piles of residue build up and obvious separation from the tape backing. It was determined that 851 is the most soluble in amyl acetate and MEK.

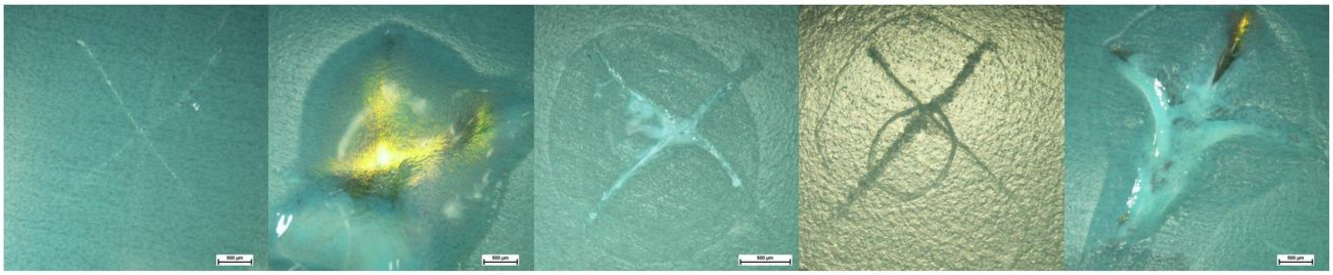


**Figure 6:** (From left to right) No solvent, amyl acetate, acetone IPA and MEK.

*7. 3M 481*

3M 481's adhesive material is rubber. Both acetone and IPA were ineffective at removing any of the adhesive. Clear X's can be seen with no stringiness or residue build up visible. MEK dissolved the adhesive a fair amount, creating a spider web effect when the X was drawn. The amyl dissolved the adhesive the best, forming a large pile of residue where the drop of solvent was applied. No X can be clearly seen, but trails of residue can. Therefore, this adhesive was most soluble in amyl acetate and MEK.

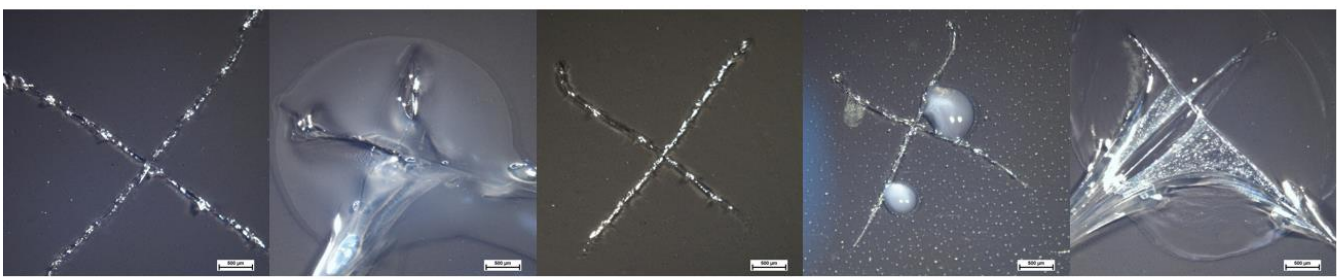


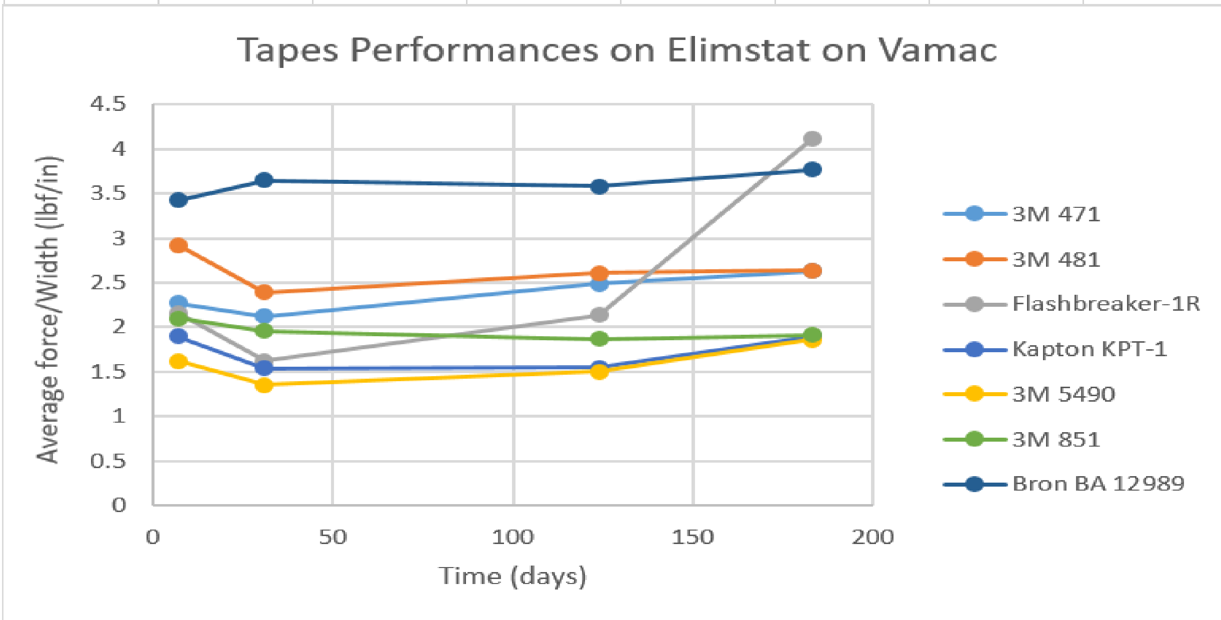
**Figure 8:** (From left to right) No solvent, amyl acetate, acetone IPA and MEK.

## B. Peel Adhesion Testing (1 month to 6 months)

The peel tests were performed simply to see how the adhesion strengths changed over time. It can be seen that adhesion strength for the tape/substrate combinations did not unanimously trend in one direction over time.

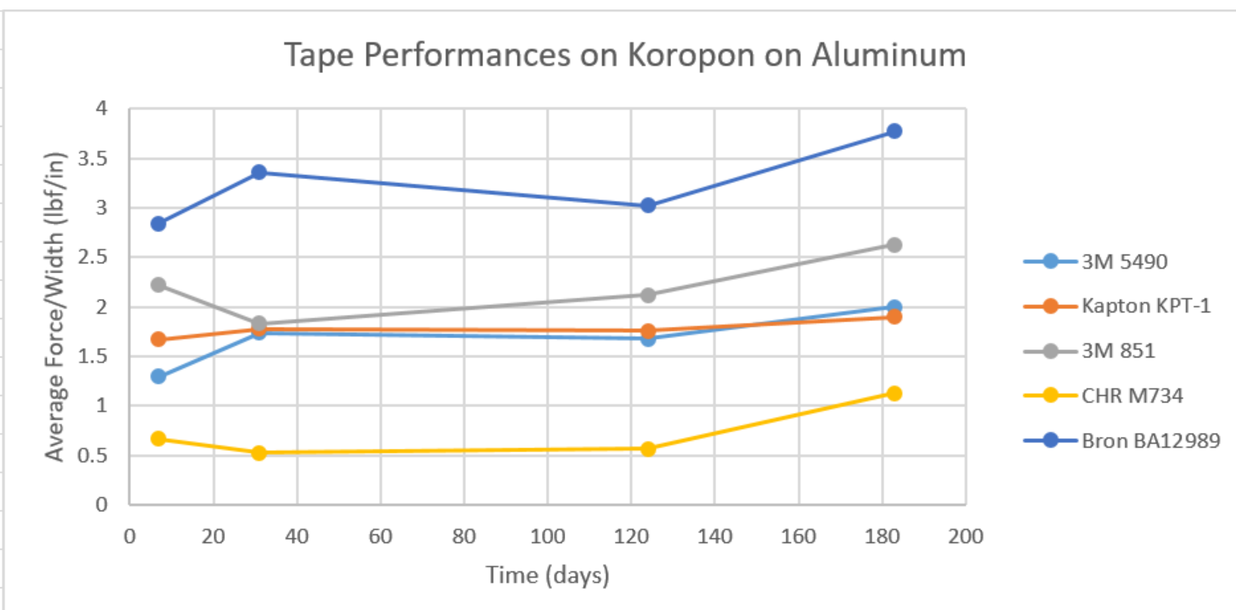
*1. Elimstat on Vamac*

| Days | 3M 471 | 3M 481 | FB-1 | 3M 5490 | Kapton KPT-1 | 3M 851 | Bron BA 12989 |
|---|---|---|---|---|---|---|---|
| 7 | 2.27 | 2.92 | 2.16 | 1.62 | 1.9 | 2.1 | 3.42 |
| 31 | 2.12 | 2.39 | 1.62 | 1.35 | 1.54 | 1.96 | 3.65 |
| 124 | 2.49 | 2.61 | 2.14 | 1.5 | 1.55 | 1.87 | 3.58 |
| 183 | 2.63 | 2.64 | 4.11 | 1.86 | 1.9 | 1.91 | 3.77 |



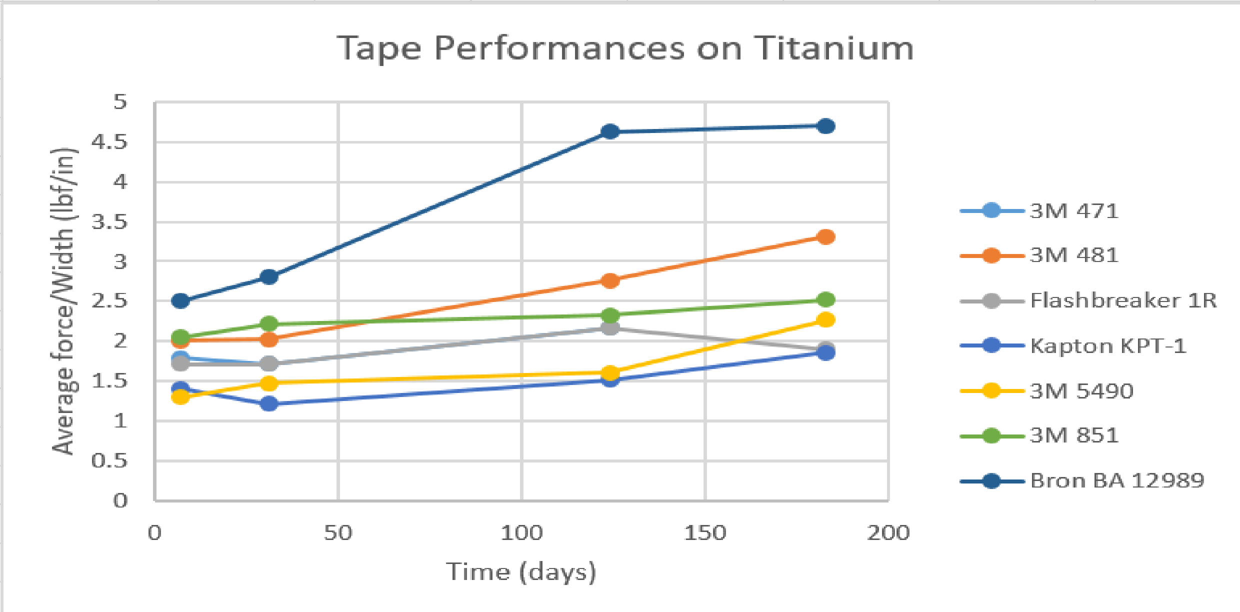

2. *Koropon on aluminum*

| Days | 3M 5490 | Kapton KPT-1 | 3M 851 | CHR M734 | Bron BA12989 |
|---|---|---|---|---|---|
| 7 | 1.3 | 1.67 | 2.22 | 0.67 | 2.84 |
| 31 | 1.74 | 1.78 | 1.83 | 0.53 | 3.36 |
| 124 | 1.68 | 1.76 | 2.12 | 0.57 | 3.02 |
| 183 | 2 | 1.9 | 2.63 | 1.13 | 3.77 |



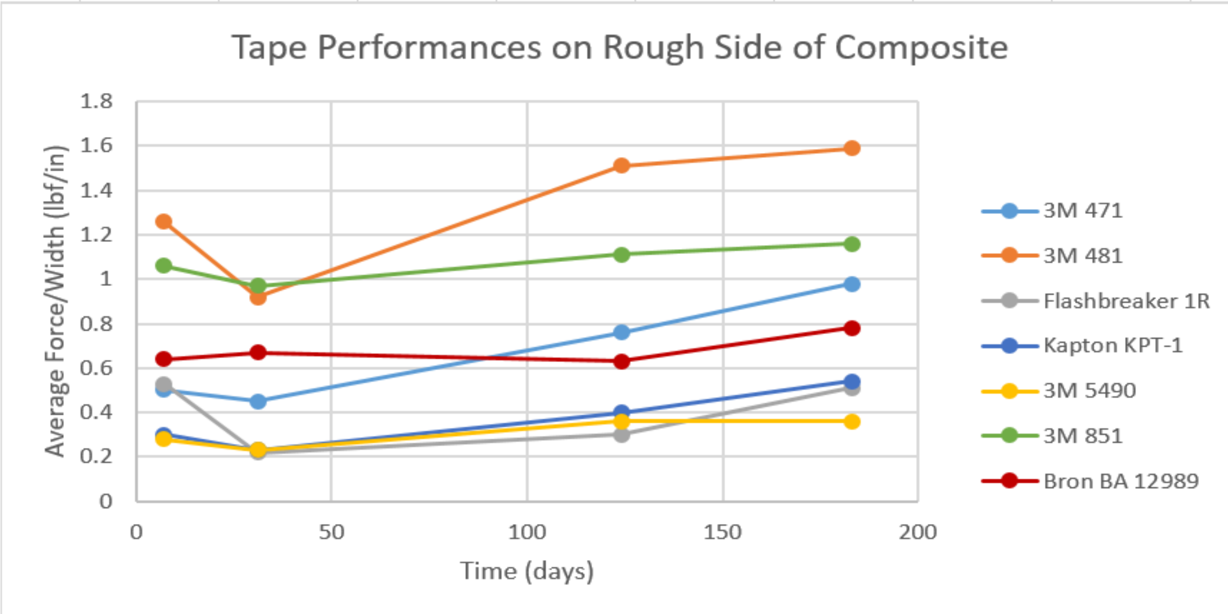
3. *Titanium*

| Days | 3M 471 | 3M 481 | Flashbreaker 1R | 3M 5490 | Kapton KPT-1 | 3M 851 | Bron BA 12989 |
|---|---|---|---|---|---|---|---|
| 7 | 1.79 | 2.01 | 1.72 | 1.3 | 1.41 | 2.05 | 2.5 |
| 31 | 1.72 | 2.03 | 1.72 | 1.48 | 1.22 | 2.22 | 2.8 |
| 124 | 2.17 | 2.76 | 2.17 | 1.61 | 1.52 | 2.33 | 4.62 |
| 183 | 2.46 | 3.31 | 1.9 | 2.27 | 1.86 | 2.52 | 4.7 |



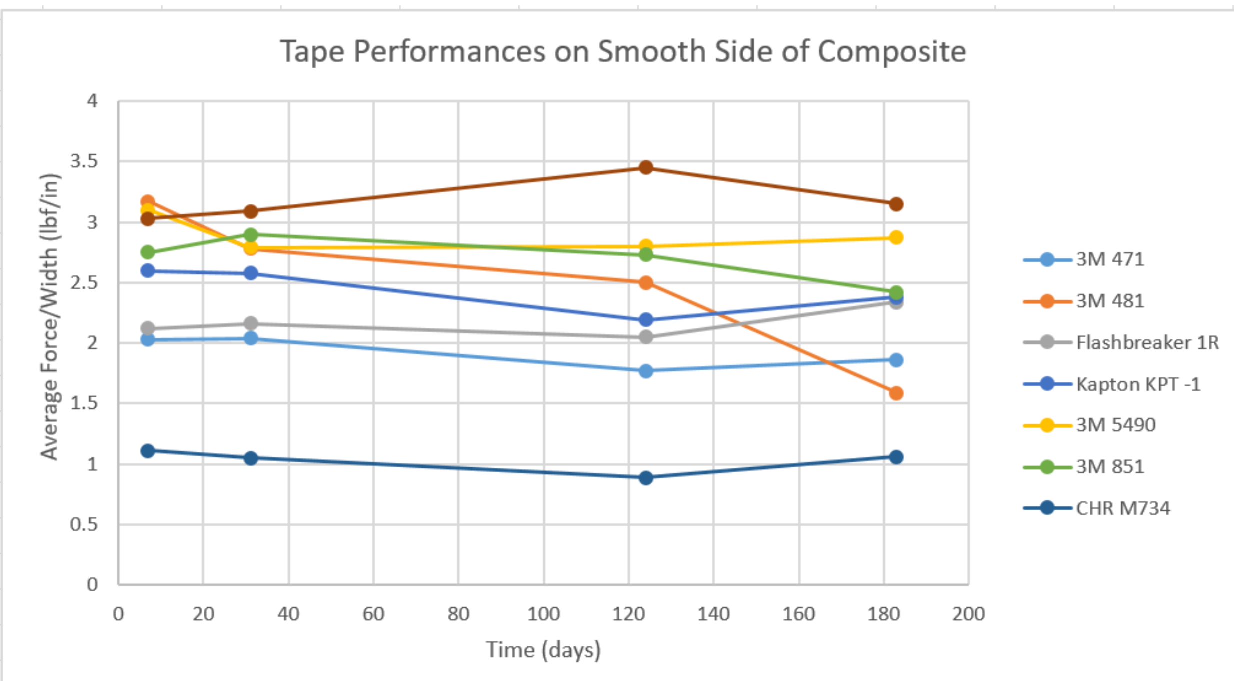

4. *Composite – Rough*

| Days | 3M 471 | 3M 481 | Flashbreaker 1R | 3M 5490 | Kapton KPT-1 | 3M 851 | Bron BA 12989 |
|---|---|---|---|---|---|---|---|
| 7 | 0.5 | 1.26 | 0.53 | 0.28 | 0.3 | 1.06 | 0.64 |
| 31 | 0.45 | 0.92 | 0.22 | 0.23 | 0.23 | 0.97 | 0.67 |
| 124 | 0.76 | 1.51 | 0.3 | 0.36 | 0.4 | 1.11 | 0.63 |
| 183 | 0.98 | 1.59 | 0.51 | 0.36 | 0.54 | 1.16 | 0.78 |




5. *Composite – Smooth*

| Days | 3M 471 | 3M 481 | FB-1 | 3M 5490 | KPT-1 | 3M 851 | CHR M734 | Bron |
|---|---|---|---|---|---|---|---|---|
| 7 | 2.03 | 3.17 | 2.12 | 3.1 | 2.6 | 2.75 | 1.11 | 3.03 |
| 31 | 2.04 | 2.78 | 2.16 | 2.79 | 2.58 | 2.9 | 1.05 | 3.09 |
| 124 | 1.77 | 2.5 | 2.05 | 2.8 | 2.19 | 2.73 | 0.89 | 3.45 |
| 183 | 1.86 | 1.59 | 2.34 | 2.87 | 2.38 | 2.42 | 1.06 | 3.15 |



### C. SEM Imaging of Titanium Substrates

The purpose of this section is to give the reader an idea of the adhesive residue that is analyzed utilizing SEM. Only the titanium coupons are included as these were finished first. The residue features are critical in determining the overall rating for a tape. The location of the excess residue as well as the degree of residue left behind all play a factor when considering the usability of a tape on the spacecraft. Only two tapes are reviewed in this section. The leading edge indicates where the tape pull began and the trailing edge indicates where the tape pull finished.

*1. 3M 471 Adhesive Residue*

In Figure 9 it can be clearly seen that the adhesive left residue behind. The outline of where the tape once was is defined and contains dark patches where clumps of adhesive remain. The tape footprint is also evident in Figure 10. On the SE image (Figure 10, right), a strip of residue can be seen where the tape pull test started. The remainder of the coupon appears to be fairly clean in comparison.

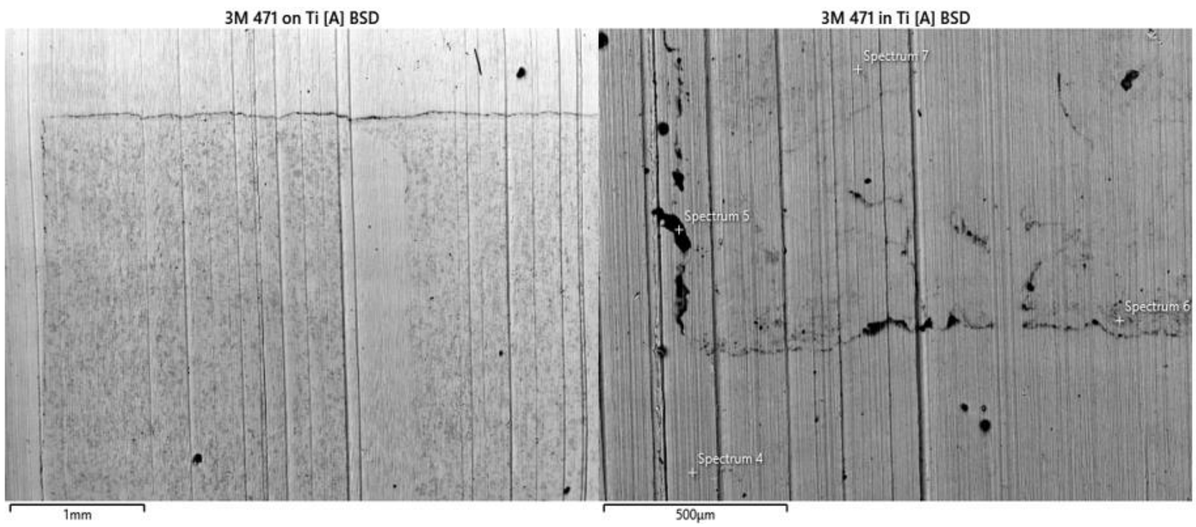



**Figure 9:** (Left) Footprint and (Right) Edge on sample A of 3M 471 on titanium.

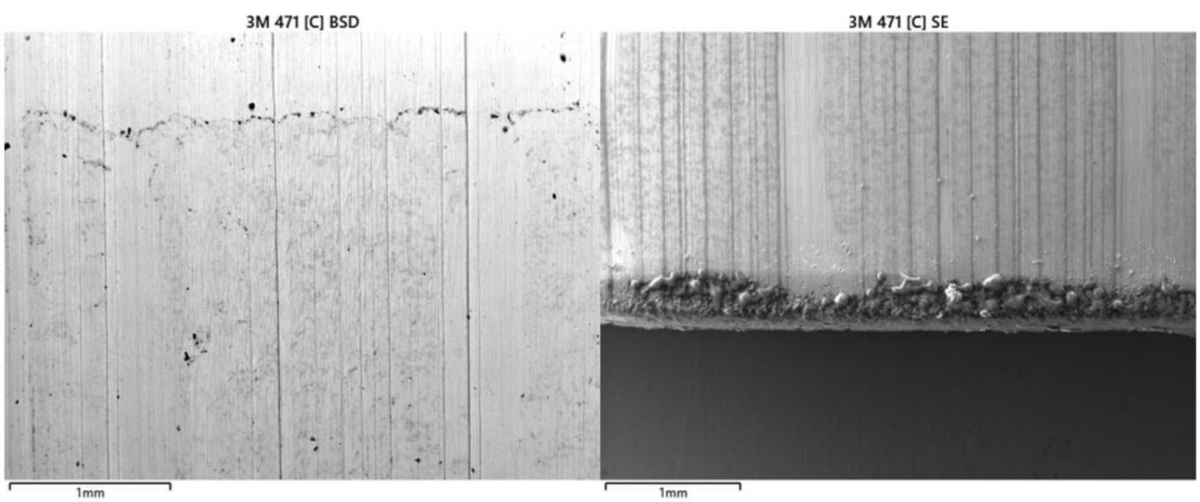



**Figure 10:** (Left) Trailing footprint and (Right) Leading Edge on sample C of 3M 471 on titanium.

*2. 3M 481 Adhesive Residue*

In Figure 11, a thick band of adhesive can be seen on the leading and trailing edge while the body of the coupon is left clean. On the leading edge of the coupon the adhesive appears to creep up the sides. However in Figure 12, the band of residue is far less severe. While clumps of adhesive still remain, it is not as concentrated as on coupons A.

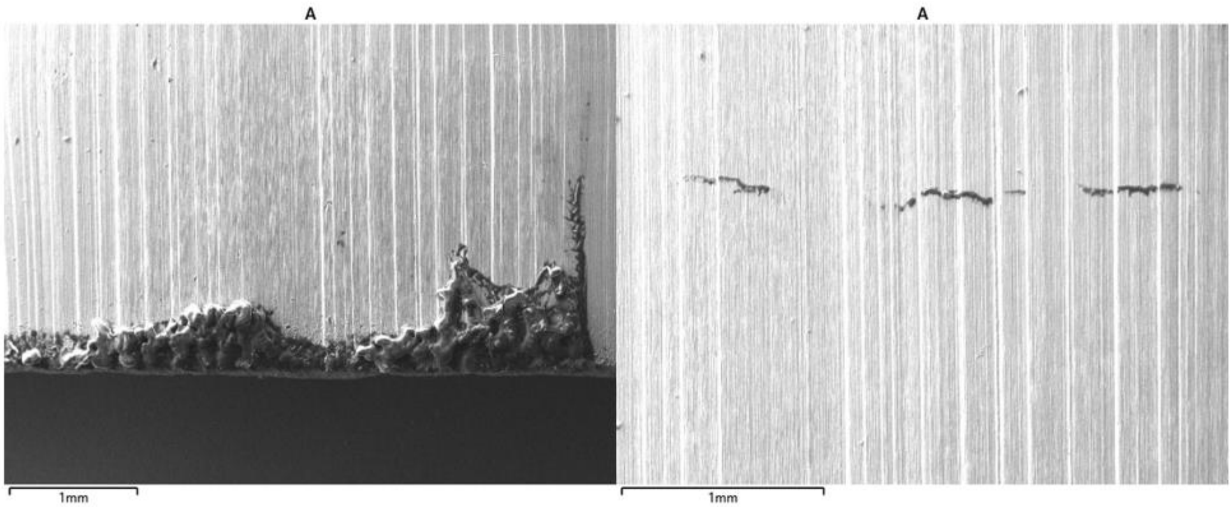



**Figure 11:** (Left) Leading edge and (Right) Trailing edge on sample A of 3M 481 on titanium.

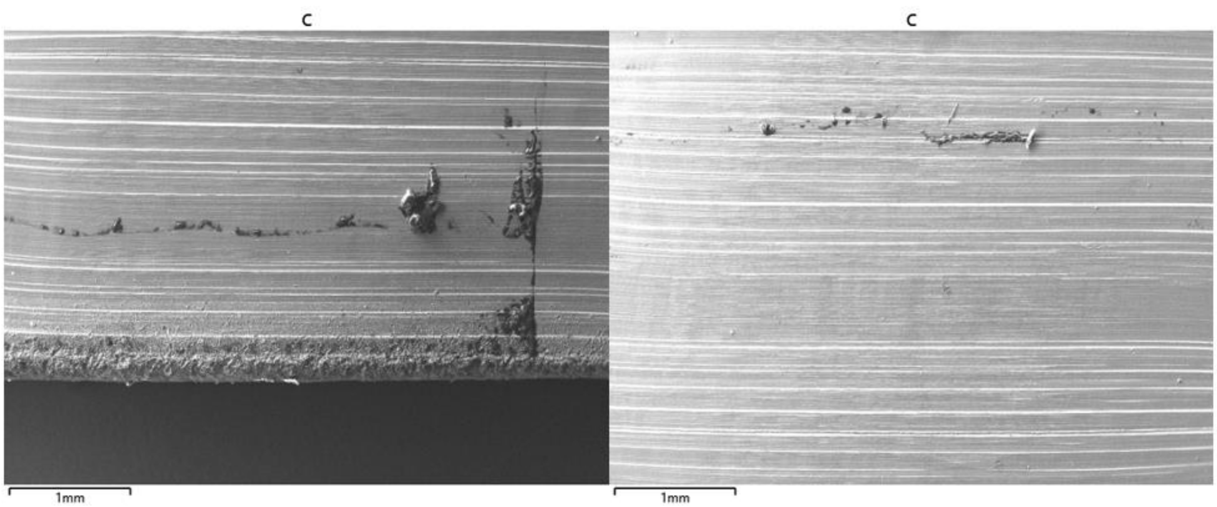



**Figure 12:** (Left) Leading edge and (Right) Trailing edge on sample C of 3M 481 on titanium.

## IV. Conclusions and Future Work

To begin, based on the data collected in this study alone, no concrete determinations can be made regarding the best adhesive to use on flight hardware substrates. However, with the experiments performed, valuable information was gained about the properties each adhesive displayed. The one acrylic adhesive, Bron BA 12989, was not completely soluble in any solvent, but could be removed marginally with either acetone or amyl acetate. The rubber adhesives, 3M 471, Flashbreaker-1R, and 3M481, responded best to amyl acetate and MEK. The rubber/silicon blend adhesive, 3M 851, was also strongly dissolved by amyl acetate and MEK. The silicon adhesives, 3M 5490 and Kapton, were found to be most soluble in acetone and MEK. The peel adhesion testing proved that none of the adhesives display a uniform progression or digression in strength when attached to different substrate materials. Moving forward, more SEM data will need to be collected to discern any trends in the residues left on the flight hardware. Based on those findings a preferred technique could be created to completely remove any residue.


## Acknowledgments

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[4] "Standard Test Method for Peel Adhesion of Pressure-Sensitive Tape," URL: http://file.yizimg.com/175706/2011120810432582.pdf [cited December 2003].