

# Impact Fatigue Performance of Adhesive Bonded Structures Based on GRIP Metal™ Concept

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## ABSTRACT

GRIP Metal™, a commercially available sheet metal product with extruded hooks, offers the potential to enhance interlaminar strength of Fiber Metal Laminates (FML) by providing mechanical fastening between the composite laminate and the aluminum sheet. However, the fatigue performance of such a joint is of concern due to the interaction between the hooks, and fibers. The purpose of this work is to evaluate the effects of GRIP Metal™ on the fatigue performance of a bonded joint using a new impact fatigue test method. Circular samples with a lap joint are fabricated by bonding two sheets of Al2024-T3 with epoxy impregnated glass fiber in between. These samples are stressed through repeated impact, for up to 600,000 cycles, to evaluate the onset of delamination and development of cracks. In addition, the effects of surface preparation including degreasing using Methyl Ethyl Ketone (MEK) only, Sol-Gel AC-130 (an adhesion promoting coating), and coating with a release agent are also studied. During impact fatigue testing, samples are inspected regularly for delamination and crack formation using an optical microscope, as well as a Scanning Electron Microscope (SEM). It was observed that samples with GRIP Metal™ contained imperfections such as cracking and delamination after approximately 140,000 cycles. It was also found that after approximately 600,000 cycles almost all samples without GRIP Metal™ had failed (complete separation of bonded structure), including those treated with surface degreasing only and most of those treated with Sol-Gel surface treatment. GRIP Metal™ samples of any hook geometry or surface treatment showed the most promising results, i.e., after 600,000 impact fatigue cycles, no separation was observed.

## 1 INTRODUCTION

Fiber-metal laminates (FML), a laminar composite of monolithic sheet aluminum bonded with fiber reinforced adhesives, have found extensive applications in the aerospace industry as a fatigue resistant alternative to monolithic aluminum. Glass laminate aluminum reinforced epoxy (GLARE), a type of FML, has in fact been incorporated into the Airbus A380 and certified for 20,000 flights with no inspection [1]. GLARE manufacturers employ a combination of chromic acid pickling and chromic acid anodizing (CAA) to create a porous aluminum oxide layer on the aluminum surface to significantly promote bonding [1]. These processes are known to be extremely harmful to the environment due to the presence and production of hexavalent chromium, which is a known carcinogen and is strictly regulated [2]. Due to this concern, these processes require extensive engineering controls and post process treatment, significantly increasing the cost of manufacturing. In this paper, GRIP Metal™ is investigated as an alternative method of surface preparation. Specifically, the fatigue bonding characteristics of GRIP Metal™ are evaluated in comparison to other surface preparations.

GRIP Metal™ made by Nucap Industries Inc. is an innovative extrusion process in which many small “hooks” are “forced out” of a metal substrate [3]. These hooks provide a mechanical interlock bonding surface which has shown significant applicability in the automotive industry as an adhesive free bonding method for backing friction “pucks” to a mounting plate in automotive brake pads [4], as shown in Figure 1.

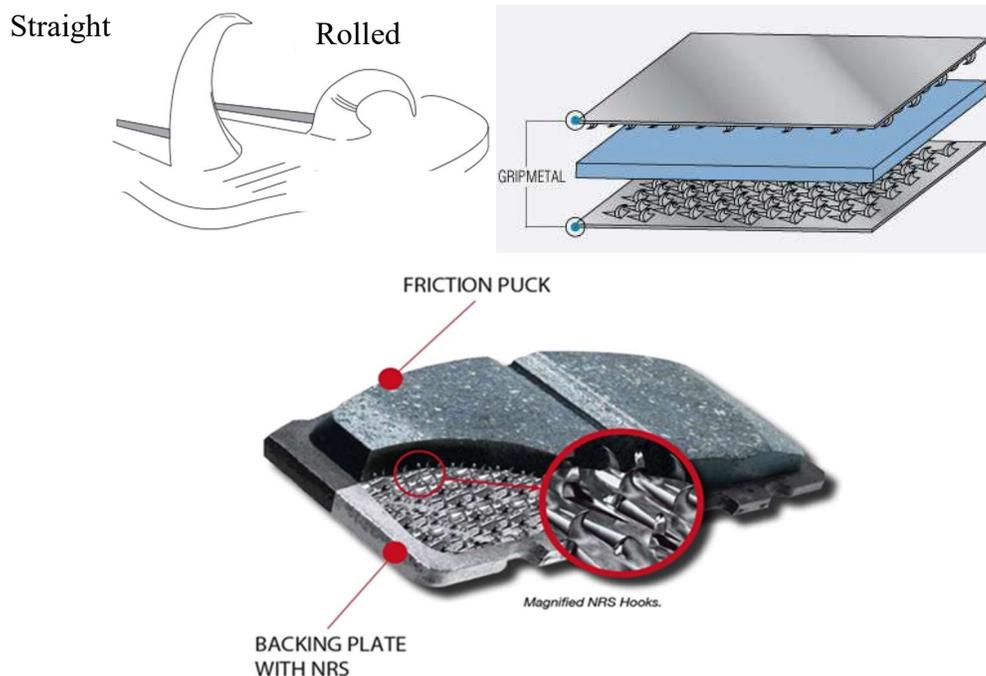


Figure 1. (a) GRIP Metal™ profile schematic [3] (b) GRIP Metal™ sandwich application concept [3]  
(c) GRIP Metal™ brake pad mounting concept [3]

One metric commonly used to determine the applicability of different types of adhesive bonding is high cycle fatigue. Currently, common practice is to use a single or double lap joint configuration in a MTS machine which produces a sinusoidal cyclic load to test the fatigue properties of an adhesive bond in accordance with ASTM D3166-99 [5]. ASTM D3166-99 recommends a cyclic loading rate of 30 Hz which means that to test one sample for 1 million cycles it would take 9.25 hours. Given the time intensive nature of this procedure, a new ranking test method was employed in this study to expedite initial feasibility and ranking tests.

## 2 METHODS

Fatigue testing of FML lap joint samples was conducted employing multiple surface preparation techniques, including different GRIP Metal™ configurations. Fatigue testing was conducted using a “tumbling” method in which samples are repetitively “dropped”. In this method, steel “scoops” running the length of the drum, bent to a 55° angle to optimize drop height at 0.356 m (14”), were riveted to the inside of a 16 Gal. (60.6 L in volume) steel drum. These “scoops” were arranged in three rows separated by 120°, and the drum was separated into three sections using two polycarbonate rings which were force fitted into ridges in the drum. This was put in place to prevent significant lateral motion of the samples. This drum was then rotated using a portable drum roller unit made by Morse Drum with a rated rotational speed of 35 RPM for the given drum size. Circular samples were then placed in the drum to be repeatedly dropped.

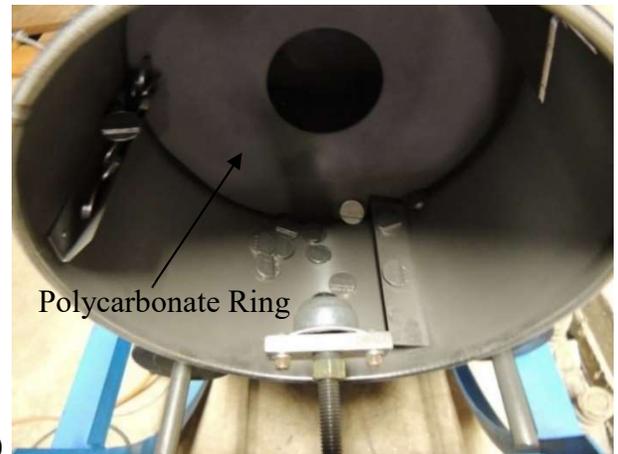
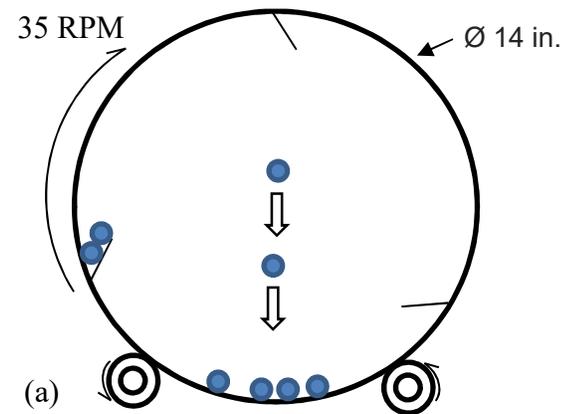


Figure 2. (a) steel drum configuration, (b) experimental test setup and (c) sample tumbling in drum.

Several different surface preparation combinations were considered for this experiment. Three types of surface configurations were employed: flat, straight hooked and rolled hooked (both of 0.04 in height). As well, three types of surface preparation techniques were employed: Loctite Frekote 770NC, ultrasonic bathing and Methyl Ethyl Ketone (MEK) degreasing, and Sol-Gel AC130 bonding primer with grit blasting. These types of surface preparations were chosen to best represent different levels of surface preparation commonly used in industry. A summary of the samples tested in this study is provided in Table 1.

Sol-Gel AC130 is a type of surface coating developed by Boeing which creates a film layer to promote organic-inorganic bonding. This coating is a water based silicon-zirconium sol-gel and is sprayed on a surface and allowed to cure naturally. Under certain conditions, Sol-Gel AC-130 is considered comparable to several anodizing processes and is used for repairs of previously anodized surfaces where anodizing is not practical [6] [7] [8]. The bonding mechanism and application method are illustrated in Figure 3.

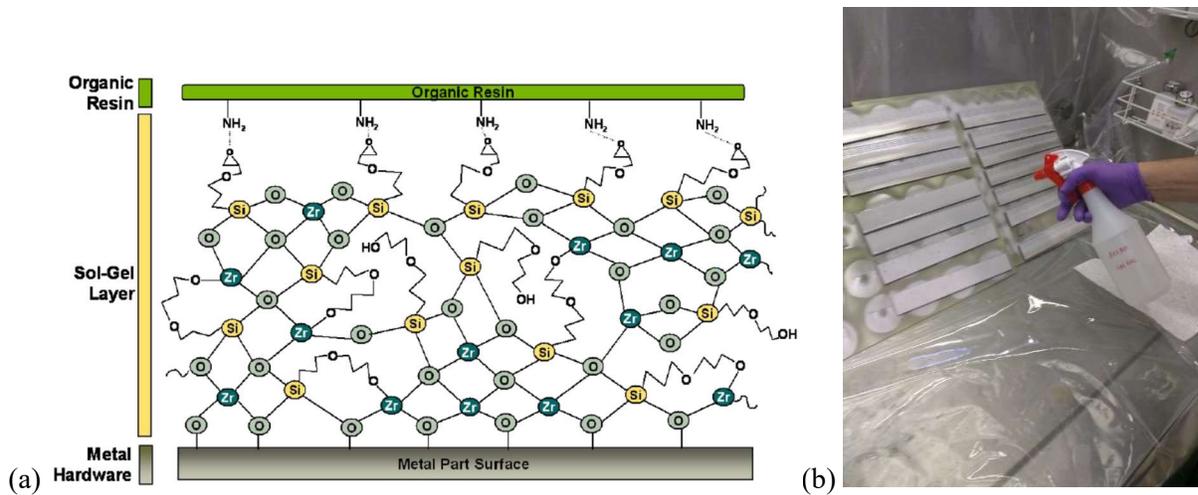


Figure 3. (a) Sol-Gel bonding layer [7] (b) Sol-Gel spray application

Sample Type	Number of Samples
Straight hooked surface with Loctite Frekote 770NC coating	18
Flat surface with MEK degreasing and ultrasonic bath	12
Straight hooked surface with MEK degreasing and ultrasonic bath	18
Rolled hooked surface with MEK degreasing and ultrasonic bath	12
Flat surface with Sol-Gel AC-130 coating	12
Straight hooked surface with Sol-Gel AC-130 coating	18
Rolled hooked surface with Sol-Gel AC-130 coating	12

Table 1. Sample Type and Sample Numbers

Circular single lap joint samples were manufactured to avoid the presence of stress concentrators. Sample specifications can be found in Table 2. These circular samples were cut from larger rectangular coupons.

The two face sheets were made of 2024-T6 aluminum with 0.04 inch hooks (GRIP Metal™ Mini) provided by Nucap and the pre-preg fiber glass reinforced epoxy was purchased from APC Composites. Material properties of this pre-preg can be found in Table 3. Aluminum shims were used to control bonding thickness, and the epoxy was cured in an autoclave, making use of vacuum bagging. Curing parameters can be found in Table 4. The circular samples were cut using water jet cutting to ensure minimal damage to the bond line.

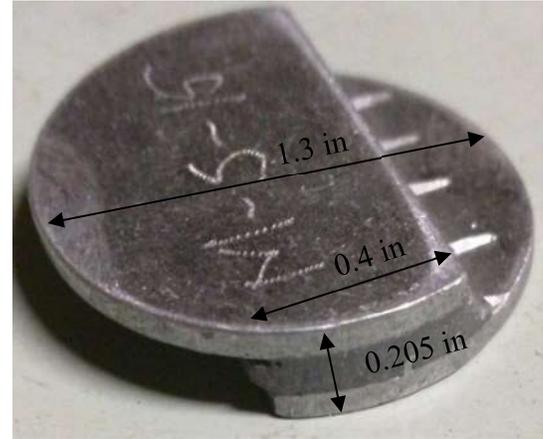


Figure 4. Test Sample

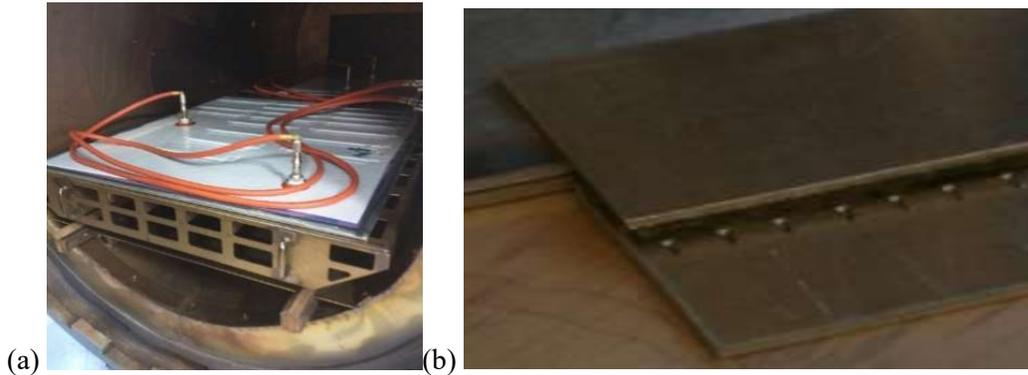


Figure 5. (a) Autoclave setup (b) lap joint coupon layup.

Pre-Preg Resin	PP-50 Thermoset Epoxy
Pre-Preg Fiber Weave	8-Harness Satin Weave
Pre-Preg Plies	12

Table 2. Pre-preg properties.

		Units
Autoclave Pressure	55	psi
Vacuum Vent Pressure	20	psi
Autoclave Temperature	305	°F
Ramp- Up Temperature Rate	5	°F/ min.
Ramp-Down Temperature Rate	5	°F/ min.
Soak Time	360	min.

Table 3. Curing Process Parameters

### 3 RESULTS

Samples were inspected utilizing visual and Scanning Electron Microscopy (SEM) over a varying interval period to detect sample failures through separation, as well as the presence of microcracks. Testing was carried out up to approximately 600,000 cycles. As interval lengths were used as a way to evaluate samples, the actual cycle to failure for each sample is in fact shorter than that when failure was detected. The inspection procedure used is summarized in Table 4.

SEM investigation was conducted by inspecting the exposed exterior edge and by dissecting samples through cross cuts to reveal the internal bond line. Dissection was done using a diamond blade saw. Once cut samples were mounted in a phenolic resin and polished to 600 grit.

When inspecting the internal and external bond line, it was discovered that regardless of hook geometry (straight or rolled) the fiber weave would bend around the hook and the hook would not penetrate the fiber weave. In other cases, the fibers appeared to have been severed around the hooks. This can be seen in Figure 6.

Upon closer inspection, it was discovered that the flat surface treated with Sol-Gel had no bond defects (Figure 7 (a)), while similarly coated surfaces with both straight and rolled hooked surfaces demonstrated occurrence of voids (Figure 8). Outside of the voids, both rolled and straight hooked surfaces with Sol-Gel appeared to have good bonding (Figure 8 (b) and (c)). It is believed that the voids were caused as a result of resin not being able to flow freely around the hooks. When examining the MEK degreased surfaces for both straight and rolled hooked surfaces, small cracks, as well as, small voids were observed (Figure 9 (b) and (c)). Frekote treated samples had small voids and cracking could be observed along the bond line (Figure 9 (a)). When looking at sample failures, all samples showed cracking of some sort on the exterior edge of the sample. It is hypothesized that this was caused impact damage to the epoxy.

The MEK flat samples all failed within 5000 cycles. Starting at approximately 375,000 cycles flat Sol-Gel samples were seen to begin failing and at approximately 550,000 cycles the Frekote hooked samples started to fail. At 600,000 cycles the Sol-Gel straight hooked samples began to fail. A summary of percent survival rate at 600,000 cycles for all samples is summarized in Figure 9 and Table 4.

Approximate Cycle Intervals	Number of Intervals	Approximate Total Cycles	Inspection Type
5,000	1	5,000	Visual
10,000	5	55,000	Visual
15,000	4	115,000	Visual
15,000	1	130,000	SEM & Visual
15,000	3	175,000	Visual
50,000	9	625,000	Visual

Table 4. Inspection Protocol

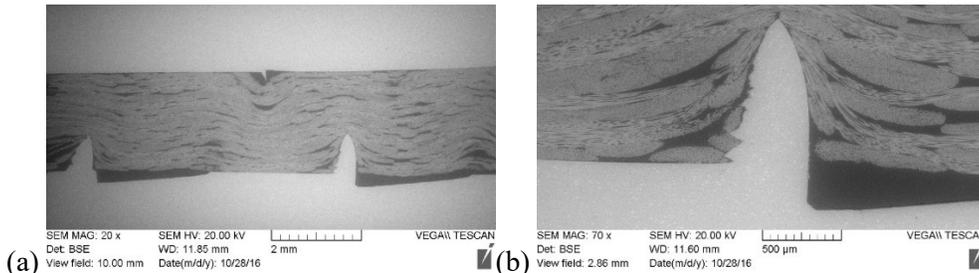


Figure 6. Bond line images (a) MEK straight hooked sample at 20X (b) MEK straight hooked sample at 70X, focusing on severed fibers.

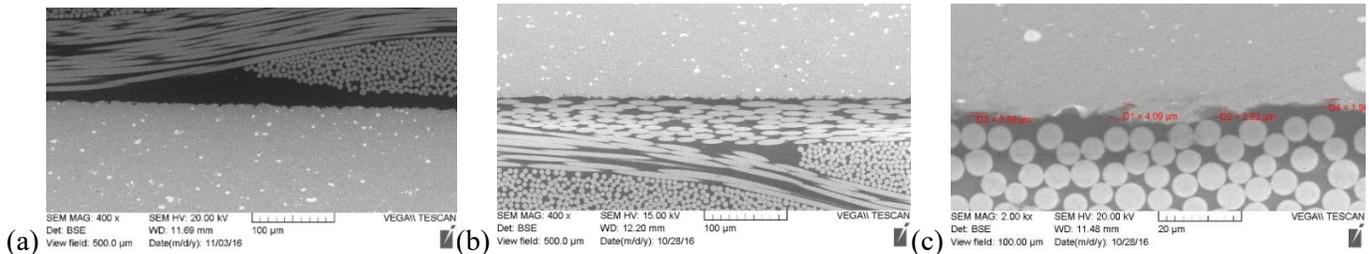


Figure 7. Bond line images (a) Sol-Gel flat sample at 400X (b) Sol-Gel straight hooked sample at 400X (c) Sol-Gel rolled hooked sample at 2000X showing coating

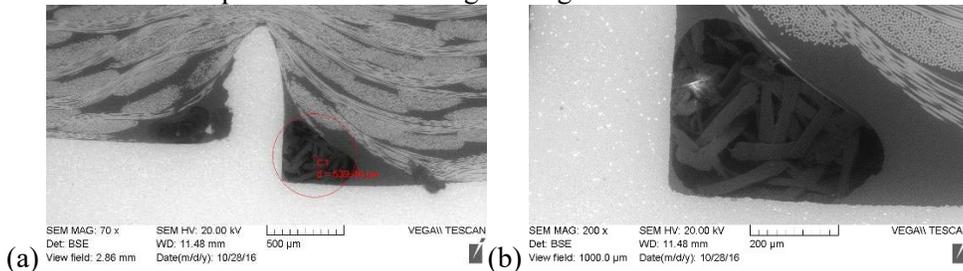


Figure 8. (a) Voiding in Sol-Gel rolled hooked sample (70X) (b) same image focusing on void at 200X

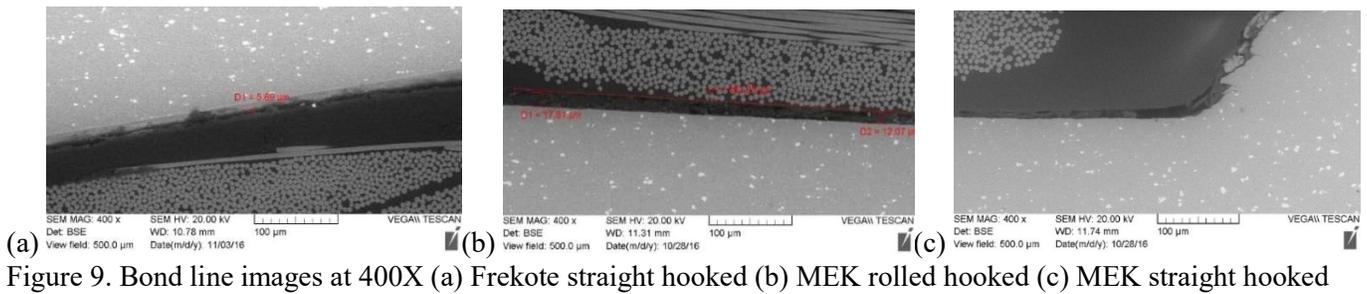


Figure 9. Bond line images at 400X (a) Frekote straight hooked (b) MEK rolled hooked (c) MEK straight hooked

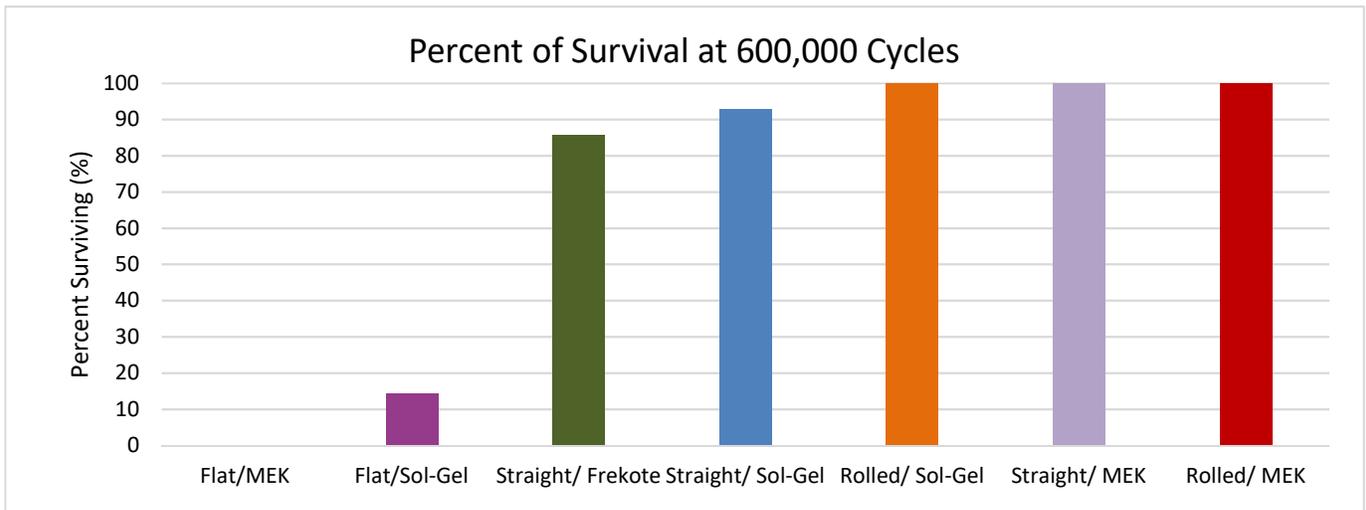


Figure 10. Percent survival of samples at 600,000 cycles

Sample Type	Survival Percentage
Flat Surface with MEK Preparation	0
Flat Surface with Sol-Gel Preparation	14.30
Straight Hooked Surface with Frekote Coating	85.71
Straight Hooked Surface with Sol-Gel Preparation	92.86
Rolled Hooked Surface with Sol-Gel Preparation	100
Straight Hooked Surface with MEK Preparation	100
Rolled Hooked Surface with MEK Preparation	100

Table 5. Survival Percentage of Samples by Type

## 4 CONCLUSION

In conclusion, this preliminary study showed that the GRIP Metal™ gives a superior advantage in enhancing the impact fatigue strength. Furthermore, with minimal surface preparation GRIP Metal™ performs significantly superior than the more elaborate surface preparation techniques applied to flat surfaces. This shows that GRIP Metal™ has its advantages in reducing the use of environmentally unfriendly processes at a lower cost. However, several shortcomings of GRIP Metal™ were observed; the inability to be fully integrated into the fiber pack and the potential damage to the fibers. As such, this technology needs further investigation to more precisely classify its capabilities, determine the effect of fiber breakage and bending during curing, and develop methods of preventing fiber breakage and bending. In future research, it is hoped that hook geometry and fiber weave configuration can be optimized so that both can be integrated.

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