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A Damage Resistance Comparison Between Candidate Polymer Matrix Composite Feedline Materials

A.T. Nettles
Marshall Space Flight Center, Marshall Space Flight Center, Alabama

National Aeronautics and
Space Administration

Marshall Space Flight Center • MSFC, Alabama 35812

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TABLE OF CONTENTS

1. INTRODUCTION 1

2. MATERIAL USED 3

3. IMPACT TESTING 4

   3.1 Visual Examination 4
   3.2 X Radiography 4
   3.3 Cross-Sectional Examination 4

4. RESULTS 6

   4.1 High-Impact Level 6
   4.2 Low-Impact Level 14

5. CONCLUSIONS 20

6. FUTURE WORK 21

REFERENCES 23

LIST OF FIGURES

1. Typical damage to impacted side of specimens at 2.5 ft-lb 7

2. Typical damage to nonimpacted side of specimens at 2.5 ft-lb 8

3. X rays of specimens impacted at 2.5 ft-lb 9

4. Cross-sectional photomicrographs of specimens impacted at 2.5 ft-lb 10

5. Cross-sectional photomicrographs of specimens impacted at 2.5 ft-lb, flourescent dye enhanced 12

6. Typical damage to impacted side of specimens at 1.8 ft-lb 15

7. Typical damage to non-impacted side of specimens at 1.8 ft-lb 16

8. Cross-sectional photomicrographs of specimens impacted at 1.8 ft-lb 18

LIST OF TABLES

1. Materials tested for impact resistance 3

LIST OF ACRONYMS

e-beam	electron beam
IM	intermediate modulus
ksi	thousand square inches
Msi	million square inches
PEEK	polyetheretherketone
PMC	polymer matrix composite
RLV	reusable launch vehicle
SARTM	solvent assisted resin transfer molding
ZnI	zinc iodide

TECHNICAL MEMORANDUM

A DAMAGE RESISTANCE COMPARISON BETWEEN CANDIDATE POLYMER MATRIX COMPOSITE FEEDLINE MATERIALS

1. INTRODUCTION

As part of NASA's focused technology programs for future reusable launch vehicles (RLV's), a feasibility study is underway of replacing metal feedlines with those made of polymer matrix composite (PMC) for transporting propulsion system liquid cryogen. The main advantage of using PMC's is in the weight reduction potential that is so critical to the next generation of RLV's. The task consists of manufacturing and testing several prototype composite feedlines made by various methods and then comparing them to see which method has the best cost and performance metrics needed. The methods used to manufacture the prototype feedlines are electron-beam (e-beam) curing, standard hand lay-up and autoclave cure, solvent assisted resin transfer molding (SARTM), and thermoplastic tape laying.

One of the critical technology drivers for composite components is resistance to foreign object impact damage. This is especially important in applications where leak paths must not develop in a component (such as a feedline). The materials examined all have a 5-harness weave of intermediate modulus 7 (IM7) as the fiber constituent (except for the polyetheretherketone (PEEK) thermoplastic, which is unidirectional tape laid up in a bidirectional configuration). The resins tested were RS–E3 (e-beam curable), PEEK, Hexcel® Incorporated 977–6, Cycom® 823, PR 520, and Shade Industries SI–SE–1, (all epoxies). Since limited material was available for impact testing, relatively small specimens were used; however, this was not a problem since the actual feedline would experience mostly contact force damage rather than damage due to large deformation. The basis for this is from a study of impact damage to composite feedlines where it was found that through the thickness leak paths developed as a result of contact force damage.[1] Impact damage resistance will be one of the critical parameters when choosing a fiber/resin system since one of the main goals of future space vehicles is increased reliability.

If possible, it would be desirable to use these feedlines without liners which add complexity and weight to the hardware. This makes permeation-after-impact testing critical to the success of the program. This type of testing is new and has not been extensively studied unlike compression-after-impact. Studies that have been performed in this area mostly pertain to using liners in composite fuel tanks.[2,3] The helicopter industry has been concerned about water ingression in honeycomb structures and has studied water permeation-after-impact.[4] Their results indicate that a liner is needed for thin facesheets. However, none of these studies examined the harsh thermal environment of cryogenic composite structures, nor do they address gas permeation through a laminate due to microcracking. Gas permeation through composites has been studied in the rocket nozzle industry since an ablative's performance is related to its permeability,[5] but this does not concern impact damage to the composite.

It is the intent of this study to gain insight into the relative resistance to microcracking due to an impact event of some candidate resins being examined for use on feedlines.

2. MATERIAL USED

There were a total of six material systems tested in this study. These, along with some of their laminate properties are presented in table 1.

All of the panels were visually inspected before test coupons were cut from them. Areas of the panels had their cross sections examined as part of another test series and no panels contained voids of any significance and all were well consolidated. Since material was limited, small impact specimens were chosen for testing. These specimens were 2.25-in. squares.

Table 1. Materials tested for impact resistance.

Material System[1]	Manufacturing Method	Type Resin	Laminate Density (g/cm^3)	Laminate Modulus (Msi)	Laminate Tensile Strength[2] (ksi)	Laminate Compression Strength[3] (ksi)
IM7/977–6	Hand Lay-Up with Autoclave Cure	Toughened Epoxy	1.35	10.4	142.0	83.9
IM7/PR 520	SARTM with Autoclave Cure	Toughened Epoxy	1.58	10.2	124.8	73.1
IM7/Cycom 823	SARTM with Autoclave Cure	Toughened Epoxy	1.63	11.7	135.3	63.1
IM7/SI–SE–1	SARTM with Autoclave Cure	Epoxy	1.55	10.2	115.0	54.2
IM7/RS–E3	Hand Lay-Up With E-Beam Cure	Epoxy	1.55	12.1	150.0	80.1
IM7/PEEK	Hand Lay-Up with Hot Press Cure	Polyetheret herketone	1.59	11.5	160.0	68.6

[1]All laminates made from 5-harness woven fabric $[0/90]_{2S}$ (Except IM7/PEEK which is made from unitape $[0,90,0,90]_S$).

[2]As specified in ASTM D 3039.

[3]As specified in ASTM D 3410M.

3. IMPACT TESTING

A drop-weight impact test apparatus was used for the testing. The square specimens were supported over a 2 × 2-in. square opening and impacted at the center with a 0.25-in. instrumented tup (striker). A few sacrificial specimens were impacted and an impact level was chosen that would produce obvious damage in most of the materials tested. This turned out to be a weight of 2.5 lbf dropped from a height of 12 in. for an impact energy of 2.5 ft-lb. This was considered the upper threshold of the impact severity and subsequent impact testing would be conducted at a level that would cause less damage. Load-time data was gathered by a GRC International Dynatup® 930–I software system for later reduction if desired. Each type of material system was impacted twice at each of the two energy levels tested to ensure repeatability. In all cases, the impacts were nearly identical in every way so repeatability was not a concern.

An impact energy level of 1.8 ft-lb was also used to observe the damage resistance of these materials at a lower severity of impact. The two impact levels used will often be referred to as high (2.5 ft-lb) and low (1.8 ft-lb) throughout this paper.

The damage resistance was evaluated in three ways: visual examination, x radiography, and cross-sectional examination.

3.1 Visual Examination

After each impact event, the specimen surface damage was recorded as a digital image. Both sides of the specimen were observed and recorded.

3.2 X Radiography

Each impacted specimen was subjected to a dye penetrant soak for at least 24 hr. The dye penetrant was a zinc iodide (ZnI) solution containing Kodak PhotoFlo™ to help the penetrant flow into all cracks and delaminations that may have formed. The ZnI solution is opaque to x rays and shows up when an x-ray image of the specimen is made, thus forming a map of the damage within the specimen. This technique was only used on specimens impacted at the high-energy level since the test at the low-energy level did not produce enough damage to be readily seen on an x ray.

3.3 Cross-Sectional Examination

After the specimens were x-rayed, they were sectioned through the center of the impact area with a diamond-wafering blade. The halved specimens were then mounted in polymethylmethacralate for subsequent edge polishing and microscopic examination. The specimens were sectioned parallel to the warp fibers on the outer surfaces of the specimens, or parallel to the outer 0° fibers on the IM7/PEEK specimens. The edges were wet polished with silicon-carbide paper of progressively finer grit sizes: 240, 400, 600, 800, 1000, and 1200. A fluorescent dye was placed on the polished edges of the specimen and wiped off so that the dye remained in any cracks in the specimen. Upon exposure to an ultraviolet light source, any damage present would then be highlighted and much easier to detect.

4. RESULTS

4.1 High-Impact Level

4.1.1 Visual Examination

Figure 1 shows the impacted side of each specimen tested at the high-energy level. In all cases, except the 977–6 resin, a distinct dent is seen in each of the specimens. Fiber breakage is observed in all of the specimens. The 977–6 resin possesses a relatively long crack across the outer warp fibers that spans the distance of the impacted zone. The PR 520 has short cracks at or near the indentation formed, as does the PEEK. The remaining three have fiber breakage within the indentation.

Figure 2 shows the back (nonimpacted) side of each type of specimen tested at the high-energy level. All samples show back face fiber breakage of varying magnitudes. The 977–6 is the least severe while the RS–E3 and Cycom 823 are the most severe where it appears near penetration occurred.

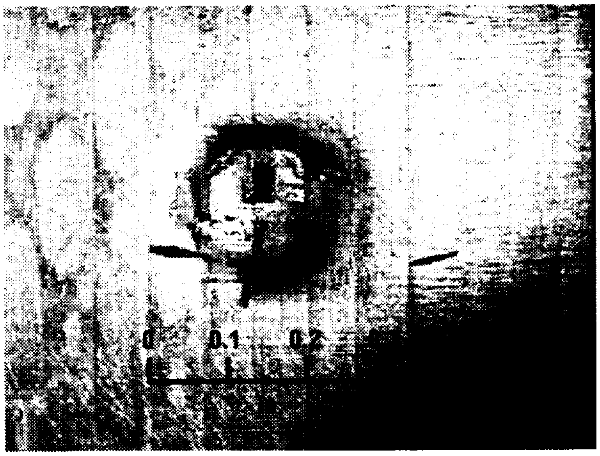
4.1.2 X Radiography

Figure 3 shows x rays of each type of resin system tested at the high-impact energy level. These x rays correspond to the visual observations of figure 1 and figure 2. The 977–6 resin system has noticeably less damage than the others. The Cycom 823 and RS–E3 systems appear as dark round circles indicating massive damage directly under the impact zone. Delaminations emanating from the impact can be seen in the Cycom 823, and RS–E3 and PEEK resin systems.

4.1.3 Cross-Sectional Examination

Figure 4 shows photomicrographs of the cross section of each type of resin system tested at the high-impact energy level. The 977–6 and PEEK resins appear to have far less damage than the others tested. The Cycom 823 and RS–E3 resins show near penetration.

Figure 5 shows fluorescent-dye-enhanced photographs of these cross sections. This technique better shows the extent of damage present in the specimens. The 977–6 specimen only has two short delaminations near the outer plies and minor fiber breakage on the top ply. The plies in the center of the specimen appear to be completely undamaged. The PEEK specimen shows good damage resistance; however, damage exists throughout the thickness of the specimen. The severity of damage to the Cycom 823 and RS–E3 resin systems are even further highlighted by the fluorescent dye. The PR 520 resin system had delamination and matrix cracking within all plies, although the photograph in figure 4 does not clearly show this and is a good argument for using a fluorescent dye penetrant even on samples where damage is readily visible.



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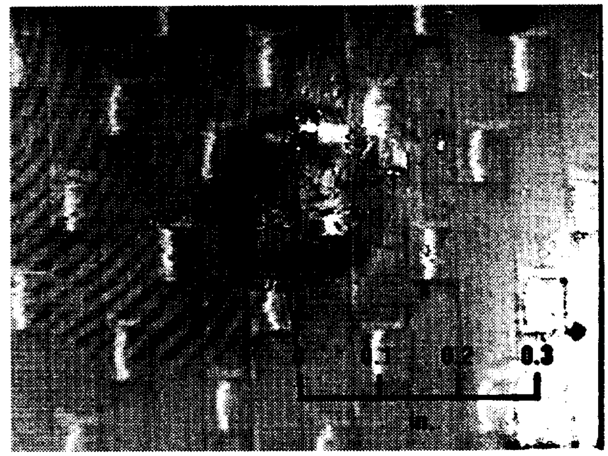

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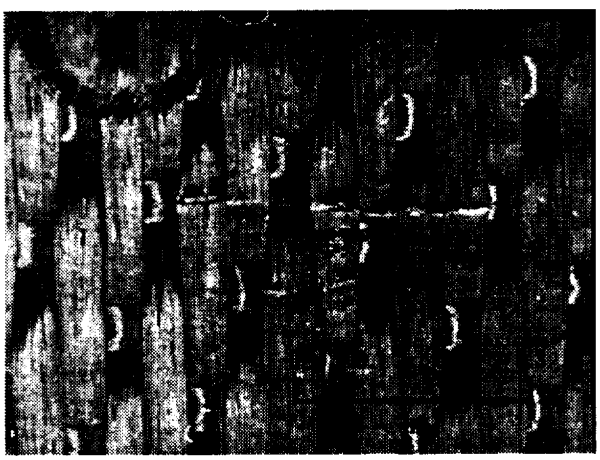

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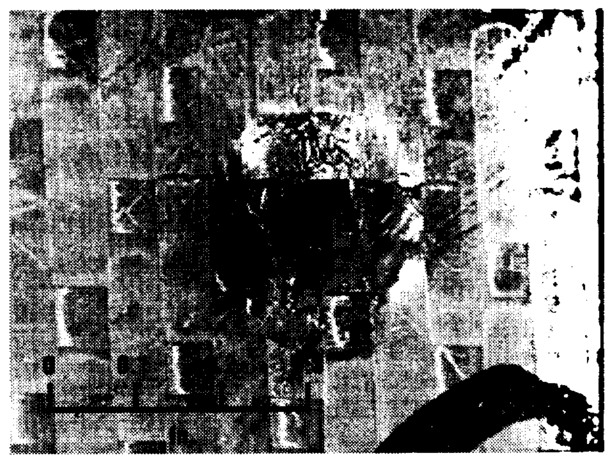

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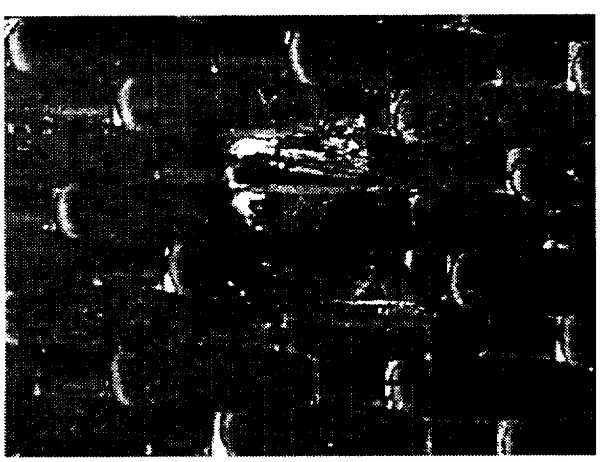
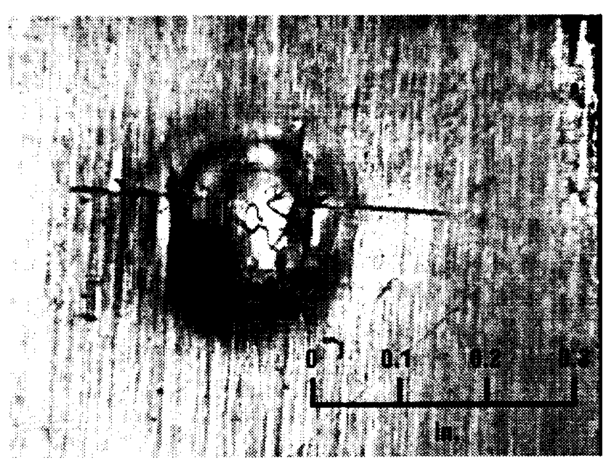


IM7/RS-E3



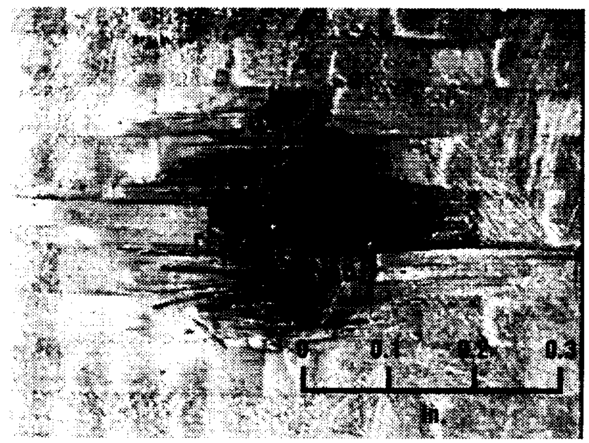
IM7/PEEK

Figure 1. Typical damage to impacted side of specimens at 2.5 ft-lb.

IM7/PR 520

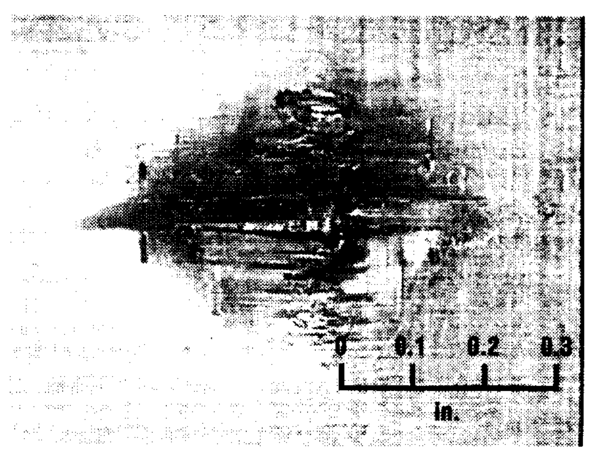
IM7/SI-SE-1

IM7/977-6



IM7/Cycom 823

IM7/RS-E3



IM7/PEEK

Figure 2. Typical damage to nonimpacted side of specimens at 2.5 ft-lb.

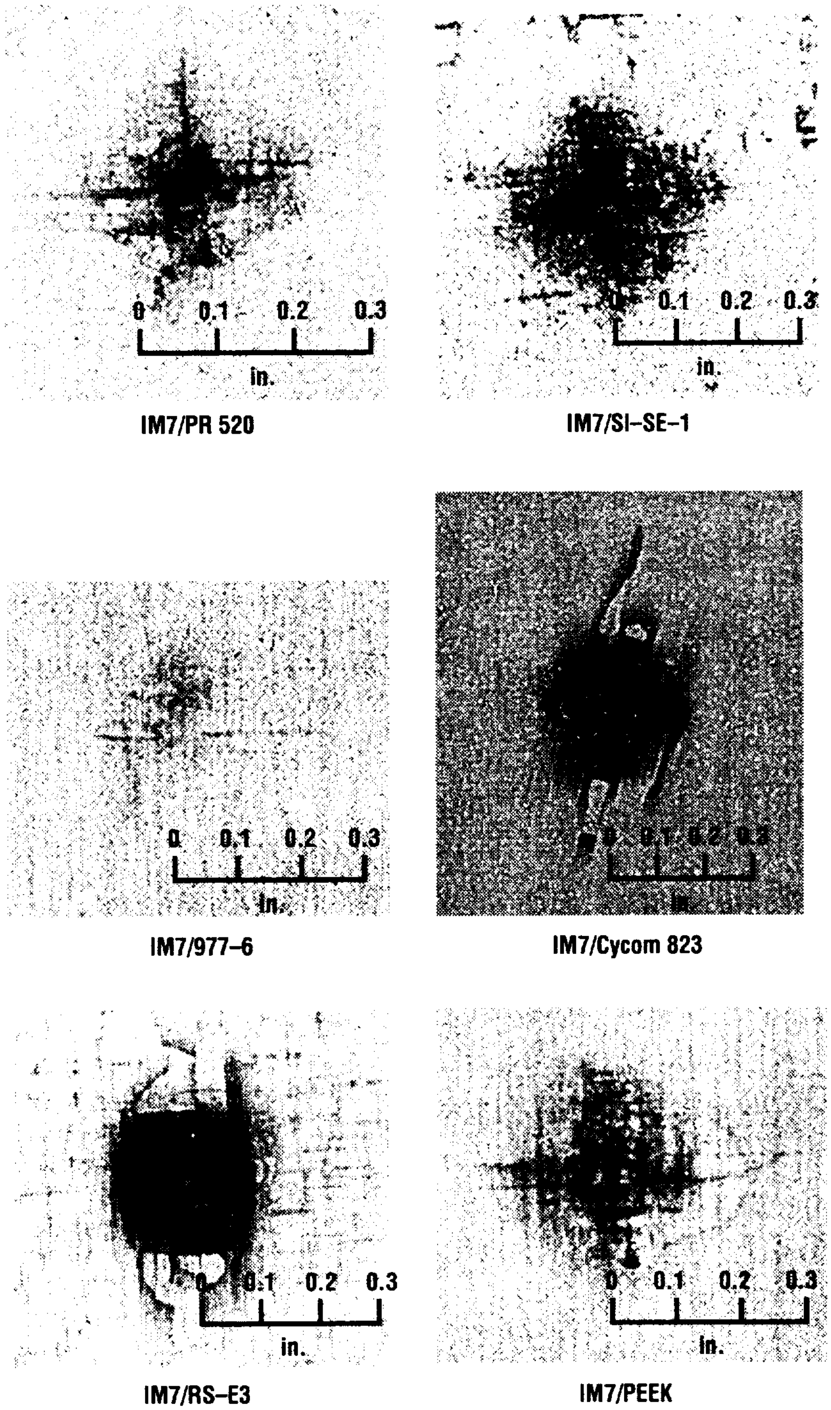
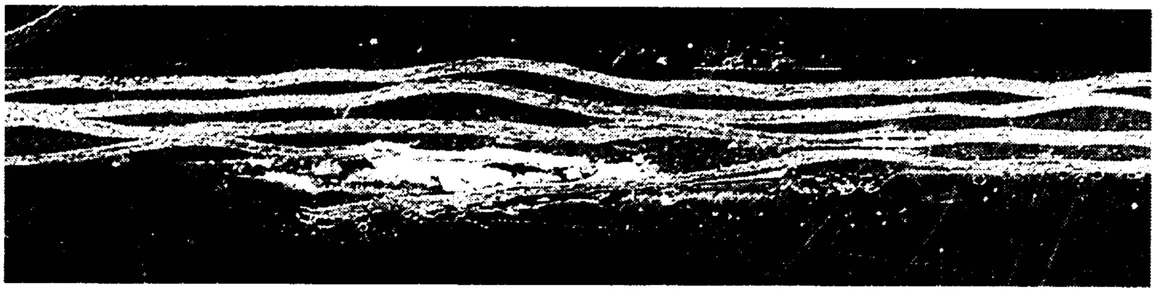


Figure 3. X-rays of specimens impacted at 2.5 ft-lb.



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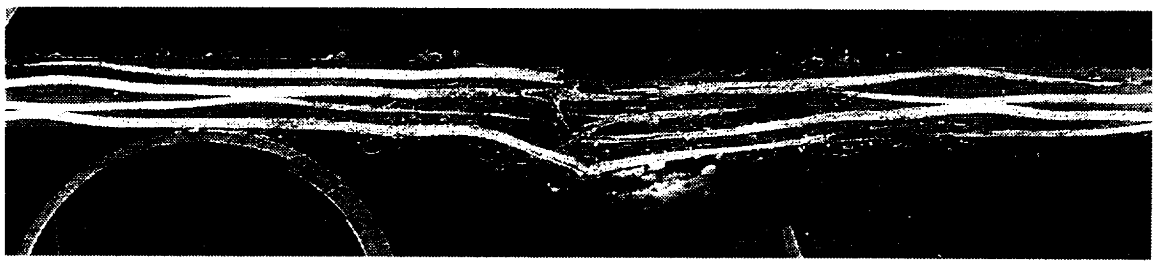

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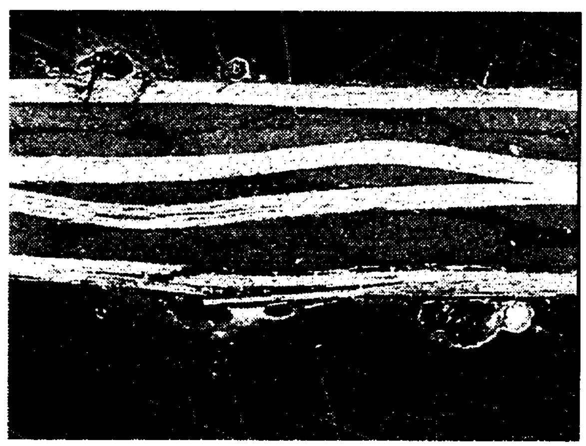
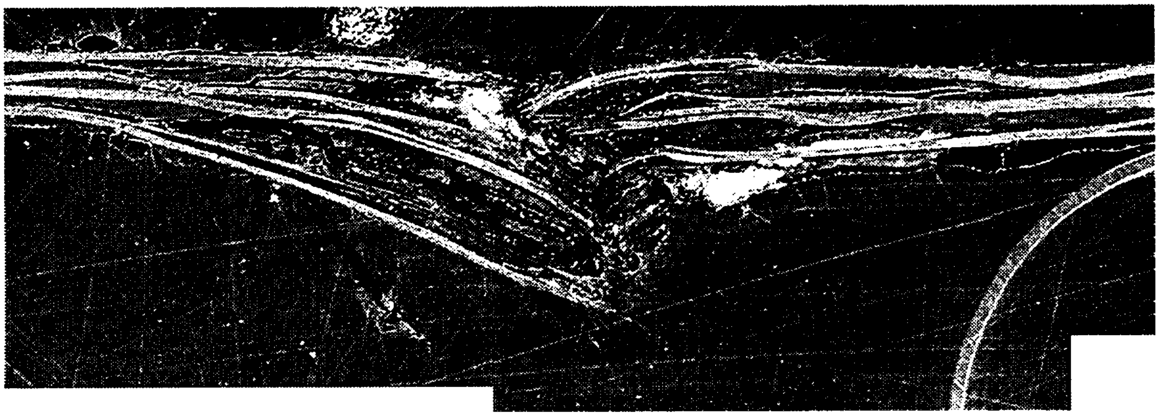


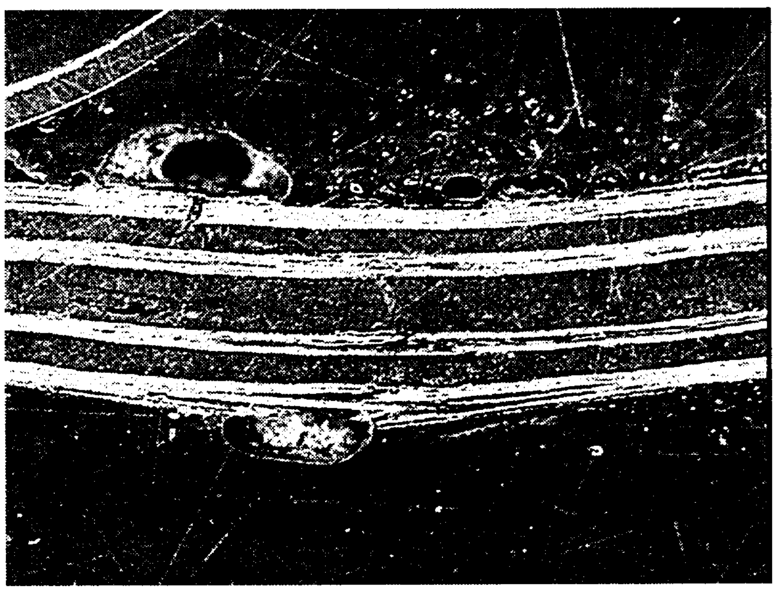
IM7/977-6

IM7/Cycom 823

Figure 4. Cross-sectional photomicrographs of specimens impacted at 2.5 ft-lb.



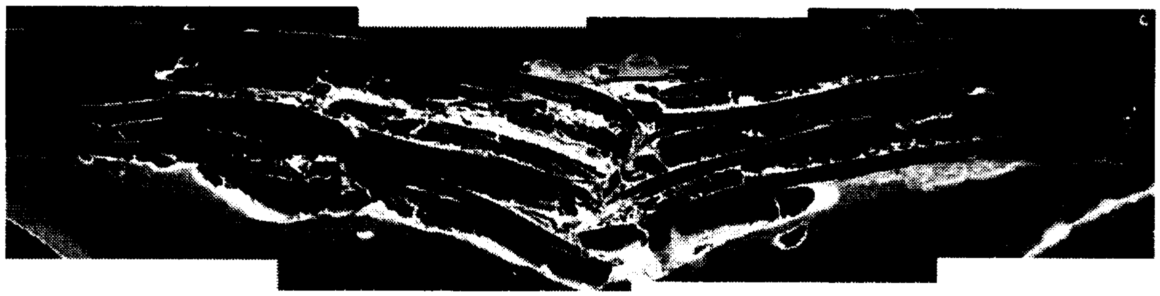
IM7/RS–E3



IM7/PEEK

Figure 4. Cross-sectional photomicrographs of specimens impacted at 2.5 ft-lb (continued).

IM7/PR 520



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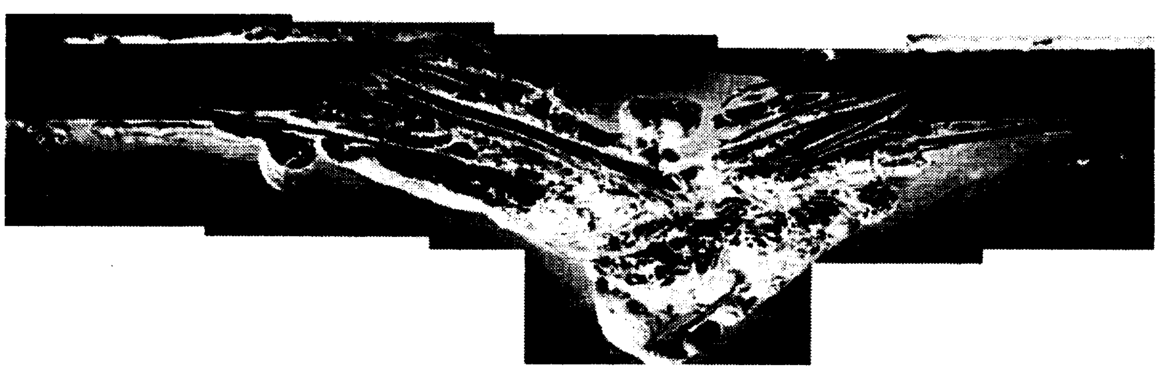
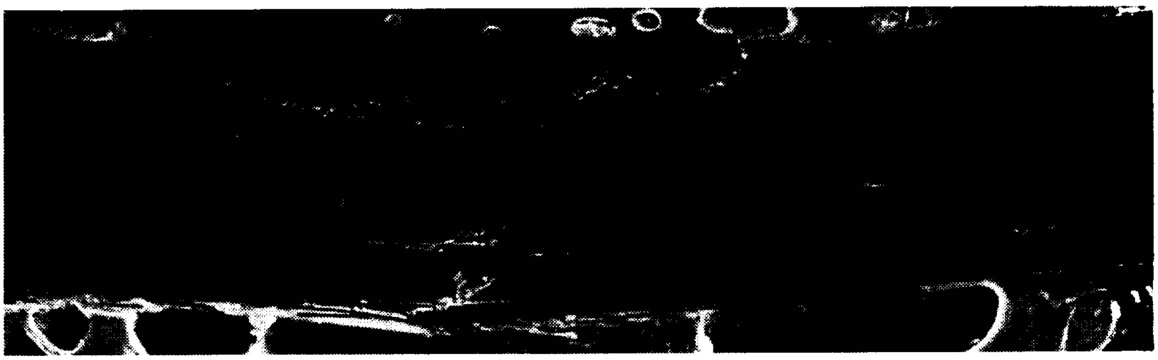
IM7/977-6



IM7/Cycom 823

Figure 5. Cross-sectional photomicrographs of specimens impacted at 2.5 ft-lb, flourescent dye enhanced.

IM7/RS–E3



IM7/PEEK

Figure 5. Cross-sectional photomicrographs of specimens impacted at 2.5 ft-lb, flourescent dye enhanced (continued).

4.2 Low-Impact Level

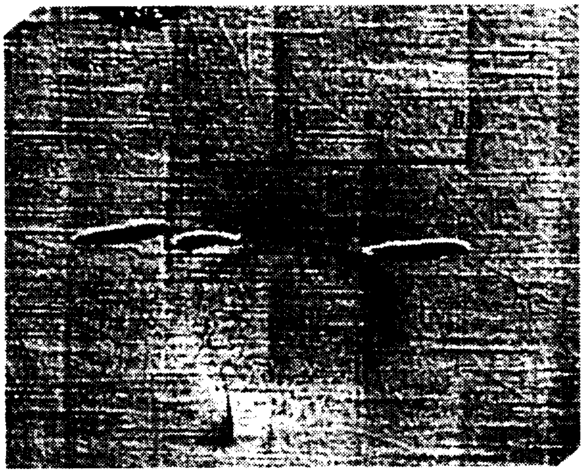
4.2.1 Visual Examination

Figure 6 shows the impacted side of each specimen tested at the low-energy level. Fiber breakage is observed in most of the specimens in the form of short cracks emanating from the indentation on the surface. The RS–E3 resin has a long, fine crack that runs from the lower left to the upper right of the figure and very little indentation is observed. The mechanics behind these small cracks is unknown, but they have been noted in a previous study.[6]

Figure 7 shows the back (non-impacted) side of each type of specimen tested at the low-energy level. All samples show back face fiber breakage of varying magnitudes. The 977–6 and RS–E3 resin systems have barely noticeable back face damage. What little damage is present in these two systems is limited to less than two fiber rows in length, but never as long as three. This type of damage could be easily overlooked during a routine inspection of a part.

4.2.2 X Radiography

No x radiography was performed for these specimens since such little damage was formed and an x ray signature of the damage would yield no information.



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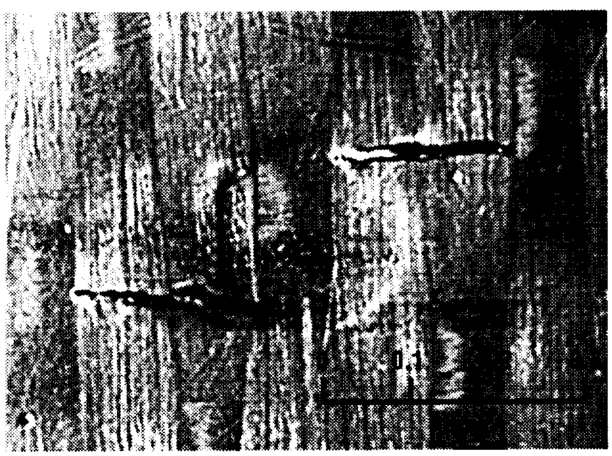

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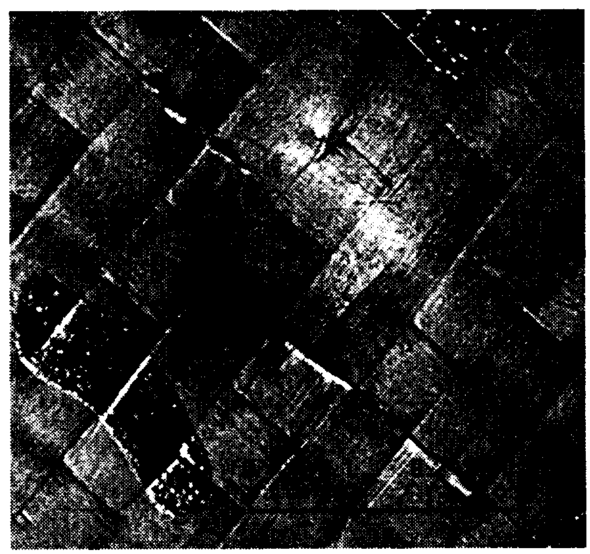

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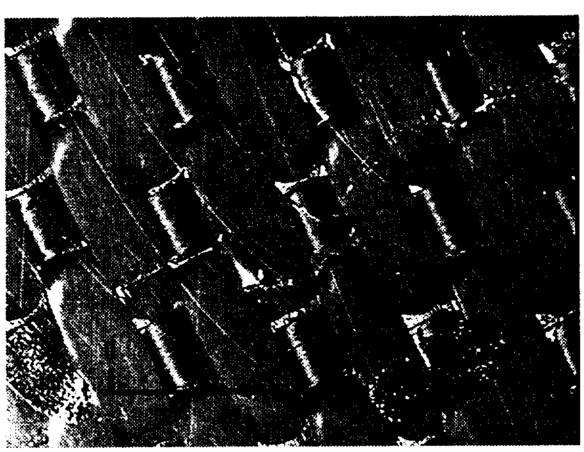

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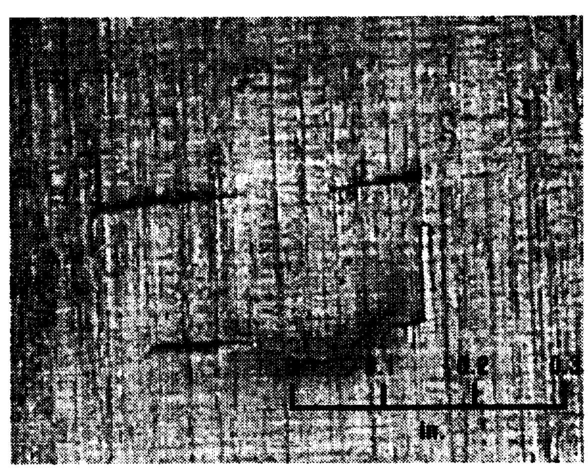
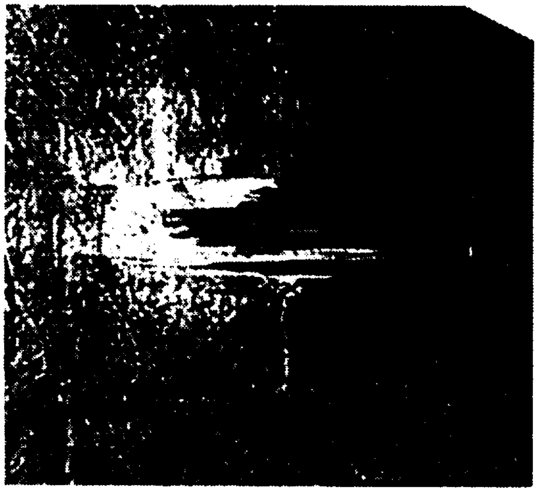


IM7/PEEK

Figure 6. Typical damage to impacted side of specimens at 1.8 ft-lb.



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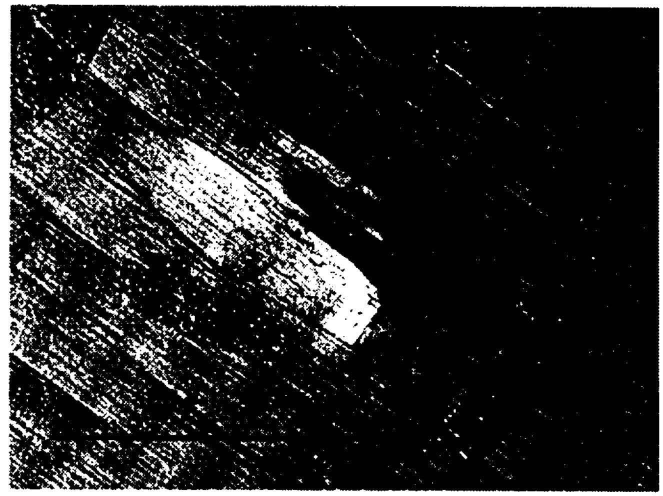

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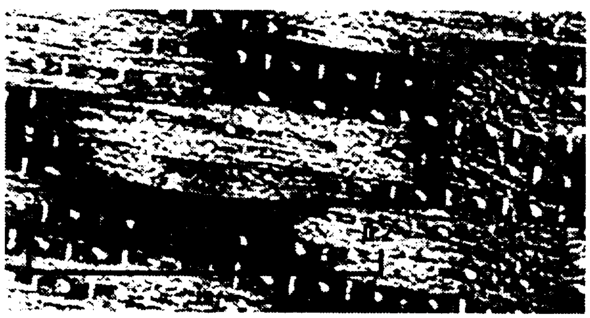
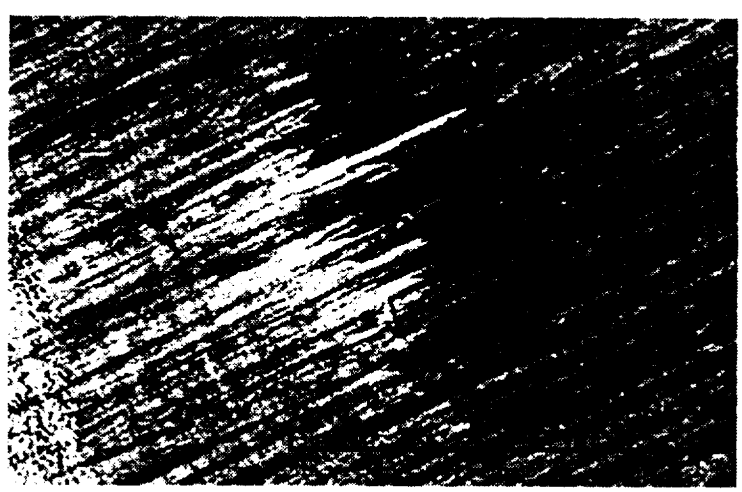


IM7/977-6

IM7/Cycom 823

IM7/RS-E3



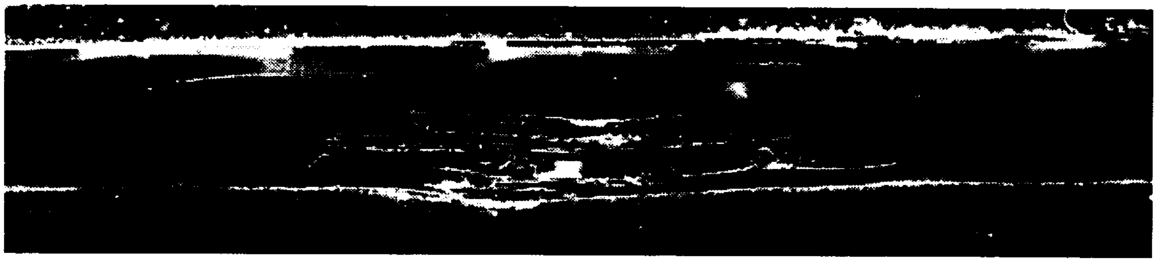
IM7/PEEK

Figure 7. Typical damage to nonimpacted side of specimens at 1.8 ft-lb.

4.2.3 Cross-Sectional Examination

Figure 8 shows photomicrographs of the cross sections of each type of resin system tested at the low-impact energy level. The fluorescent dye technique was employed on all of these specimens since visible light did not readily detect all of the damage present. The 977–6 and the PEEK resins showed the least damage as they did for the high-impact energy level. The 977–6 resin showed a short delamination at the bottom plies and some matrix cracking within the bottom ply, but the majority of the cross section was undamaged. The PEEK resin showed similar damage with some small delaminations and matrix cracking in or near the bottom ply.

The RS–E3 resin system had extremely long delaminations emanating from the impact damage, so two pictures are presented to include the entire range of damage.



IM7/PR 520

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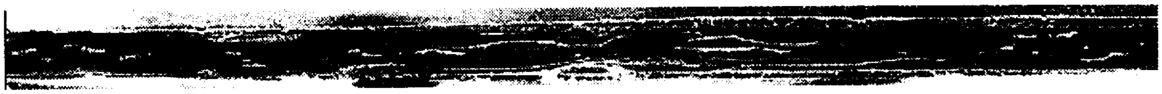
IM7/977-6

IM7/Cycom 823

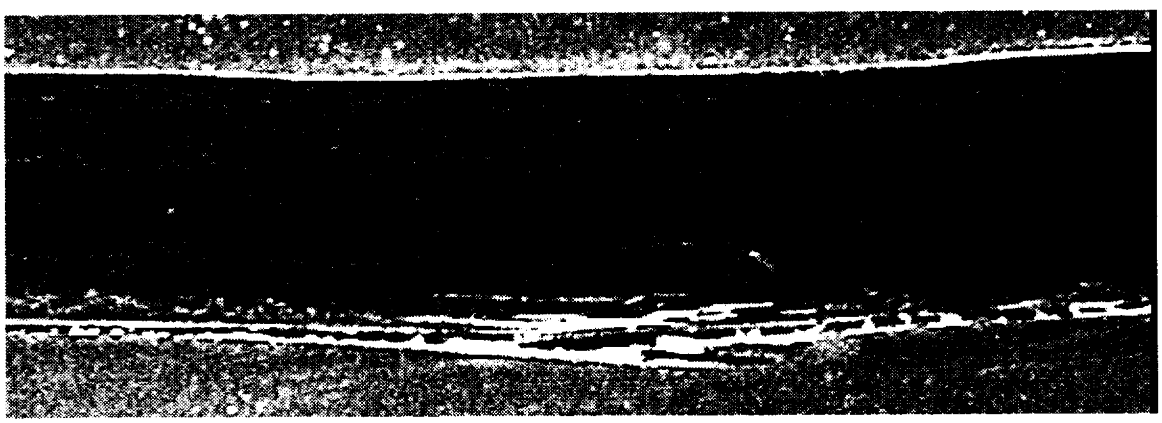
Figure 8. Cross-sectional photomicrographs of specimens impacted at 1.8 ft-lb.



Far View

Close View

IM7/RS-E3



IM7/PEEK

Figure 8. Cross-sectional photomicrographs of specimens impacted at 1.8 ft-lb (continued).

5. CONCLUSIONS

From the results in this study, it appears that the 977–6 resin system is far superior to the others tested for microcracking resistance to impact damage. The PEEK resin system also demonstrated good impact resistance. The Cycom 823 and RS–E3 demonstrated poor impact resistance as many delaminations and microcracks were found in these specimens at the low-impact energy level.

The use of a fluorescent dye to highlight damage gives a better indication of damage when examining the cross section of an impacted specimen.

6. FUTURE WORK

The next step in this damage tolerance study is to perform permeability testing of impacted samples. While cross-sectional examination gives a good idea of the relative resistance to impact damage of a resin system, resistance to permeability after an impact event is really the driver behind choosing a resin system for ducts and feedlines.

Correlation between permeability rate and microcracking as observed in cross sections will be made to gain a qualitative assessment of visual damage to permeability rates.

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As part of NASA's focused technology programs for future reusable lauch vehicles, a task is underway to study the feasibility of using the polymer matrix composite feedlines instead of metal ones on propulsion systems. This is desirable to reduce weight and manufacturing costs. The task consists of comparing several prototype composite feedlines made by various methods. These methods are electron-beam curing, standard hand lay-up and autoclave cure, solvent assisted resin transfer molding, and thermoplastic tape laying. One of the critical technology drivers for composite components is resistance to foreign objects damage. This paper presents results of an experimental study of the damage resistance of the candidate materials that the prototype feedlines are manufactured from. The materials examined all have a 5-harness weave of IM7 as the fiber constituent (except for the thermoplastic, which is unidirectional tape laid up in a bidirectional configuration). The resin tested were 977–6, PR 520, SE–SA–1, RS–E3 (e-beam curable), Cycom 823 and PEEK. The results showed that the 977–6 and PEEK were the most damage resistant in all tested cases.

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