Title: *Investigation of the Leak Response of a Carbon-Fiber Laminate Loaded in Biaxial Tension* for Proceedings of the **American Society for Composites—**
Twenty-Eighth Technical Conference

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ABSTRACT

Designers of pressurized structures have been reluctant to use composite materials because of concerns over leakage. Biaxial stress states are expected to be the worst-case loading condition for allowing leakage to occur through microcracks. To investigate the leakage behavior under in-plane biaxial loading, a cruciform composite specimen was designed that would have a relatively large test section with a uniform 1:1 biaxial loading ratio. A 7.6-cm-square test section was desired for future investigations of the leakage response as a result of impact damage. Many iterations of the cruciform specimen were evaluated using finite element analysis to reduce stress concentrations and maximize the size of the uniform biaxial strain field. The final design allowed the specimen to go to relatively high biaxial strain levels without incurring damage away from the test section. The specimen was designed and manufactured using carbon/epoxy fabric with a four-ply-thick, quasi-isotropic, central test section. Initial validation and testing were performed on a specimen without impact damage. The specimen was tested to maximum biaxial strains of approximately 4500με without apparent damage. A leak measurement system containing a pressurized cavity was clamped to the test section and used to measure the flow rate through the specimen. The leakage behavior of the specimen was investigated for pressure differences up to 172 kPa.

INTRODUCTION

Significant concerns exist regarding the ability of composite structures to contain gases as successfully as metallic designs. Flow paths through the structure may be created from linked microcracks and delaminations [1-10]. The leakage of gases may result in not meeting structural requirements or structural failure [1]. Microcracking can be created by thermal [1-3], mechanical [4-6], or combined loadings [7-9]. In addition, low-energy impacts have been shown to result in sufficient damage to allow leakage to occur [11-12]. After potential leak paths develop, the applied loads affect the opening of the cracks and, hence, the leak rate. Biaxial tensile loading appears to be the worst-case

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condition for opening the leak paths. Most pressurized structures have multi-axial loading as a result of the internal pressure (e.g., a pressurized cylinder has both hoop and axial stresses). Consequently, there is a need to develop test methods to measure leakage with biaxial tensile loads applied to the test specimen.

Biaxial tensile specimens have been previously designed and tested to obtain material properties for both metals and composites. The simplest and most common specimen is a cruciform where each arm is gripped and loaded along two orthogonal axes. Biaxial test frames are commercially available to apply these loads. Many designs for the cruciform specimen have been proposed and analyzed for metallic [13-14] and composite specimens [4,15,16]. The designs range from uniform thickness to variable thickness with several thickness reductions going from the grips to the central test section. The designs also use various corner geometries or slots to reduce stress concentrations and maximize the region of uniform biaxial strains. In all cases, the specimen needs to be designed and sized for the load frame giving consideration to grip locations, grip geometry, and force capacity of the loading axes. In reference [9], a flat uniform-thickness "tetra-axial" specimen with eight loading arms was developed that used four loading axes to obtain the desired strain field.

In reference [15], finite element analyses were used to analyze representative composite cruciform specimens reported in the literature. It was found that true biaxial fields are not produced in the gage sections due to large strain concentrations produced by the geometry. An improved cruciform specimen was proposed but was not manufactured. Despite improvements to the design, the modified specimen still had high values of shear strain in the outer fillets. Specimens with and without slots were analyzed and optimized in reference [14]. The slots were found to improve the uniformity of the strain distribution in the central gage area.

Several techniques are used for assessing the leakage. The simplest method uses a leak detection fluid applied to the unpressurized surface that gives a visual indication of leakage. However, most investigations require a quantitative measurement. The selected technique is typically dependent on the gas, pressure differential, and the flow rate of interest. To measure very small flow rates, a mass spectrometer is often used in conjunction with helium. For slightly larger flow rates, a method consistent with ASTM D1434 is used where a manometer containing a "slug" of fluid on the unpressurized surface is monitored [17]. For larger flow rates, flow meters and pressure controllers can be used [10-11].

To measure the leak rate, a fixture must be sealed to the specimen to create a pressure differential between the opposing sides. Typically, a metal fixture containing a cavity is sealed to the specimen using gaskets or O-rings. For uniaxial testing, a relatively wide specimen is required to accommodate the fixture. For uniaxial tests of four-ply carbon/epoxy laminates, the permeability was found to be nearly constant up to strains of 6000$\mu\varepsilon$ [6]. A biaxial-loaded specimen must also have a relatively large region where the biaxial strains are uniform. This typically requires a customized design that includes a larger test region and an allowance for a flat clamping surface for the leak measurement fixture. A cross-ply, cruciform specimen that allows for leakage testing was developed in reference [4]. Using a helium leak detector, the leakage was reported to increase "remarkably" under biaxial loading for laminates containing impact damage. This specimen contained a 100-mm-diameter region of uniform thickness. However, the specimen was not analyzed to determine the properties of the central strain field. The "tetra-axial" specimen was also successfully used for leak testing [9].

An alternate technique that is used to obtain a biaxial strain field and simultaneously investigate the leak behavior is through the use of large (up to diameters of 64 cm) pressurized circular disks [5,8]. However, leak testing of pressurized disks has a number of limitations. To achieve the desired biaxial stress state, very high pressures are often used. These pressures typically exceed the expected pressure differential for the application. The high pressures often create high strains around the perimeter that may fail the specimens. Consequently, the highest strain may not be the biaxial strain that is desired in the center of the specimen. If a specimen were to fail, a sudden depressurization may injure personnel and damage equipment. The leakage rate is also very difficult to measure during a pressurized disk test. Typically, a fluid is sprayed on the exterior surface to visually look for leaks. This creates additional safety concerns and only qualitative results are obtained. In addition, some of the fluid is absorbed by the test specimen, which limits flow or may even seal the specimen. Consequently, this leakage detection method may yield non-conservative results.

The objective of this work was to design a new composite cruciform specimen, manufacture the design, and then perform tests to validate the design and investigate the leakage behavior. A specimen design was needed that had a large area of uniform biaxial strain that would accommodate the damage from an impact. A specimen configuration is required that exhibits a 1:1 biaxial strain state over a 7.6-cm-square gage section that will be involved in the leakage test. The design is also required to avoid any failures outside the gage area at biaxial strain strains of at least 4000με. The test section was selected to be a four-ply quasi-isotropic laminate made from IM7/977-2 4-harness satin-weave fabric. Due to the coarse woven architecture, fabric plies are believed to be more susceptible to leakage than tape plies. The geometry of a fabric ply creates the potential for a large number of leak paths between the fiber bundles that a biaxial stress could open. Four plies of fabric are expected to be the minimum gage used on most structures due to structural property and damage resistance considerations. In general, the susceptibility to leakage increases with decreasing laminate thickness. IM7/977-2 has been reported to be more resistant to microcracking and leakage than other material systems [2]. The specimen was manufactured and installed in an existing biaxial test frame.

In this paper, the design, manufacture, and testing of a new composite cruciform specimen is described. A design study was conducted using finite element analysis based on the specimen requirements. The design of the composite specimen was based on a successful metallic cruciform that was designed and sized for the existing biaxial test machine [13]. The strain concentrations in the new design were minimized by changing both the layup and specific geometric features. After an acceptable design was reached, a test article was manufactured for evaluation. Some modifications to the specimen were required for alignment purposes. The performance of the specimen was then assessed under biaxial loading with and without applied pressure. Finally, the leak response was investigated as a function of the average biaxial strain using a pressurized flow measurement system [11] that attached to the specimen.

LEAK MEASUREMENT SYSTEM

The leak rate was measured in standard cubic centimeters per minute (sccm) using the pressurized flow measurement system depicted in Figure 1 [11]. The system simultaneously controlled the pressure at a specific value while measuring the flow rate. This system was developed for a previous test program, and additional details can be found in reference 1. The self-contained system consists of flow meters, pressure/flow controllers, readouts, and valves (Figure 1). Bottled nitrogen was used for the testing. The test specimen was clamped between two aluminum fixtures containing a 7.6 x 7.6 x 0.64 cm cavity (Figure 2) and attached to the leak measurement system using a high-pressure hose. The specimen was sealed to the fixture using a gasket and high-pressure vacuum grease. The pressure controller is used to maintain the pressure that is set on the system. In order for the pressure controller to operate, a small leak rate is initially set using a microvent valve before the specimen is pressurized. After the flow rate for this small initial leak rate has stabilized, the value of the flow rate is recorded. A valve is then opened to pressurize the specimen and the flow rate is allowed to stabilize again. The measured flow rate after the valve is opened to the specimen is compared to the initial flow rate to obtain the flow (or leakage) rate through the specimen. Differential pressures up to 172 kPa were applied to opposite sides of the specimen. Due to small fluctuations in the flow rate, this system was not able to measure leakage rates below 1 - 2 sccm.

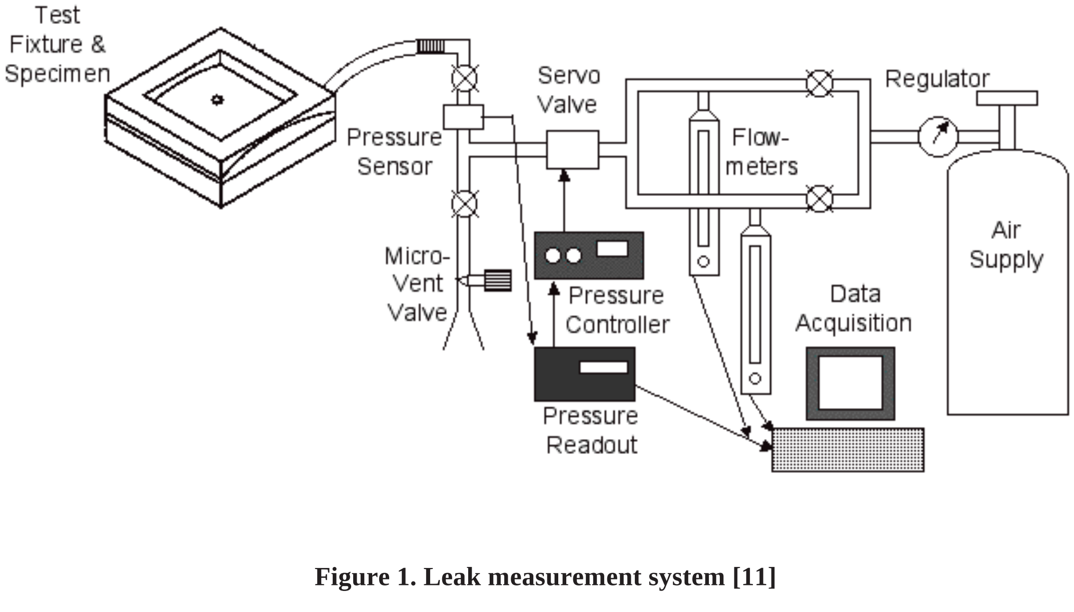

Figure 1. Leak measurement system [11]

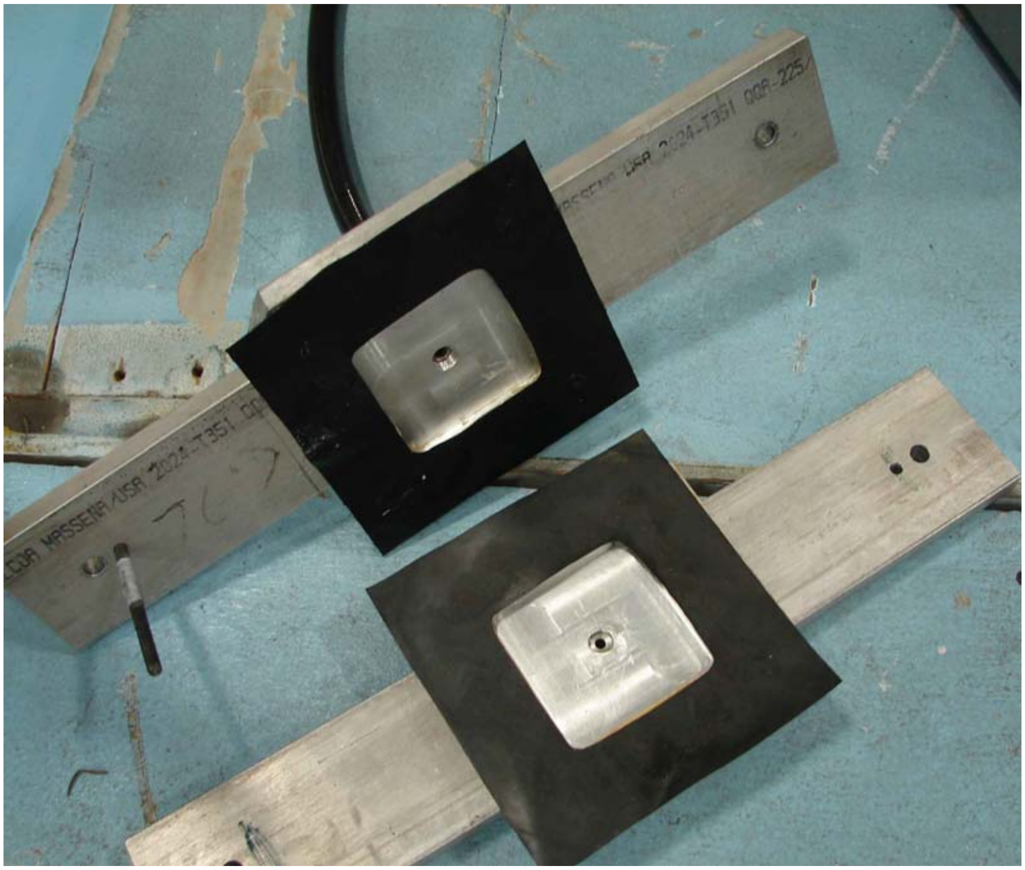

Figure 2. Open pressurization fixture showing rubber gasket and cavity

BIAXIAL TEST MACHINE PROCEDURES

The specimen was tested in an existing biaxial test facility located at NASA Langley Research Center (Figure 3). This planar biaxial test system uses four hydraulic actuators, four load cells, and four mechanical grips to apply the loads to a cruciform specimen. The opposing pairs of actuators had maximum load capacities of 445 and 667 kN. Each grip has five large bolts arranged in two rows that go through matching holes in the loading arm of the specimen. A single multi-channel digital controller was used to simultaneously control all four actuators. The movements of all four actuators are displacement controlled and linked together such that the specimen remains centered in the test frame.

Figure 3. Biaxial Test Machine

The alignment of the cruciform specimen with the actuators and the grips is critical to achieving the desired deformation of the specimen and preventing premature damage during loading. The four actuators are attached to the square load frame by pinned joints at each corner. An alignment procedure had been previously developed for metallic specimens, and a similar procedure was used for the composite specimen. This procedure involved installing a high-tolerance alignment pin through the central hole of each grip and the matching hole of each loading arm of the specimen. The composite specimen is fairly flexible and could be easily damaged by eccentric or out-of-plane loads. Consequently, the specimen was first pinned along diagonals to maintain alignment and prevent damaging loads. To align the actuators relative to the specimen, forces of approximately 20 kN were applied to the actuators while in displacement control with the controller software keeping the specimen centrally located in the frame. With the alignment force applied, four bolts were installed in the remaining bolt holes in each grip and tightened to a specific torque value. The last step involved replacing the central alignment pin in each grip with a bolt.

From previous biaxial testing experience on metallic specimens, it was determined that eight three-gage rosettes are required on a cruciform specimen to ensure proper alignment. A pair of rosettes was bonded onto opposing front and back surfaces at each of the four corners (location details will be shown later). The alignment was checked by recording the strain behavior of all gages while the actuator forces were increased from approximately 4 to 90 kN. For an acceptable alignment, the percent bending for each of the back-to-back gages at the corners is required to be less than 5%. The slopes of the force-strain curves for gages with identical orientations were also required to be within 5%. If the alignment was found to be unacceptable, the specimen was completely removed then reinstalled. Prior to leak testing, the specimen was installed in the test frame several times to refine the installation procedure, train personnel, and check the specimen alignment.

BIAXIAL TEST SPECIMEN DESIGN

This section will describe the design and analysis of the composite, biaxial, cruciform specimen. The material selected for this study was a 4-harness satin weave fabric made from IM7/977-2 and manufactured by Cytec Industries. The test area was specified as four-layers of fabric with a quasi-isotropic layup, which was expected to be the minimum gage that would be used on a pressurized composite structure. The specimen was designed for a 1:1 biaxial stress ratio with a central region of relatively (±8%) uniform biaxial strain to attach a leakage measuring system. The goal of this investigation was that leakage be measured while the composite material system is held at a 1:1 biaxial state of strain of at least 4000$\mu\varepsilon$. While the test section is held at this strain level, the design should not allow areas away from the center to develop damage, which was assessed by utilizing a maximum strain criterion during the design analyses discussed in this section.

To this end, a cruciform specimen was designed for installation into the existing grips and testing in the biaxial test facility at NASA Langley. The specimen was based on a successful metallic design [13] that is used at this facility and illustrated in Figure 4. The metallic cruciform was designed using 2D finite element analysis to maximize the region of uniform biaxial strain while reducing stress concentrations away from the test area [13]. A somewhat unique feature of the metallic design was a slotted region in each loading arm. The 12 long slots (illustrated in Figure 4), located just outside of each grip, are used to direct and align the load to the center of the specimen, which prevents the load from “bypassing” the test section. The slots accomplish this by increasing the cross-section compliance. The main stress concentrations are located at the fillets and at the end of the slots. In addition to geometry changes to reduce these concentrations, the thicknesses in these areas were increased. Since this design was optimized for metallic materials, the initial composite cruciform design was based on this geometry.

Several different permutations of the composite, biaxial specimen were examined using finite element analysis. Although the slotted design is typically avoided for composite cruciform designs, the slots were included to obtain a more uniform biaxial strain field. However, the number of slots was reduced relative to the metallic design. A finite element mesh of one of the specimen permutations analyzed is shown in Figure 5. The specimen consists of three main sections: a circular, central gage section; an intermediate section (rounded square section surrounding the gage section); and a slotted outer section, the ends of which are gripped in the biaxial test fixture. The stacking sequences (used in the final specimen design) of these three sections are included in Figure 5.

All the finite element models consisted of four-node shell elements (ABAQUS element type S4), and one quarter of the specimen was analyzed on the assumption of quarter symmetry. A composite layer material model was employed to represent the stacking sequence of each section of the specimen (the material properties utilized to represent the composite material are given in Table 1). The cured ply thickness was assumed to be 0.206 mm. Load application was modeled through the application of prescribed displacements at the rows of nodes connected to the arrows illustrated in Figure 5. A typical model size included approximately 130,000 degrees of freedom (length of isoparametric elements in gage section was 3.6 mm). All analyses were performed using ABAQUS/Standard version 6.7-2. Various stacking sequences for the three main sections depicted in Figure 5 were considered in the analyses. In addition, the dimensions (including width, length and slot radius) and number of the slots were varied. The analyses were used to determine the state of biaxial strain in the central gage section and the maximum and minimum stress and strain values developed in the specimen. A maximum strain criterion was employed to judge the likelihood of specimen failure on the basis of first-ply failure considerations. Overall specimen response was also computed to check if a given specimen design could be loaded to the required state of biaxial strain without exceeding the capacity of the load frame. The configuration that achieved the desired specimen response corresponds to that shown in Figure 6.

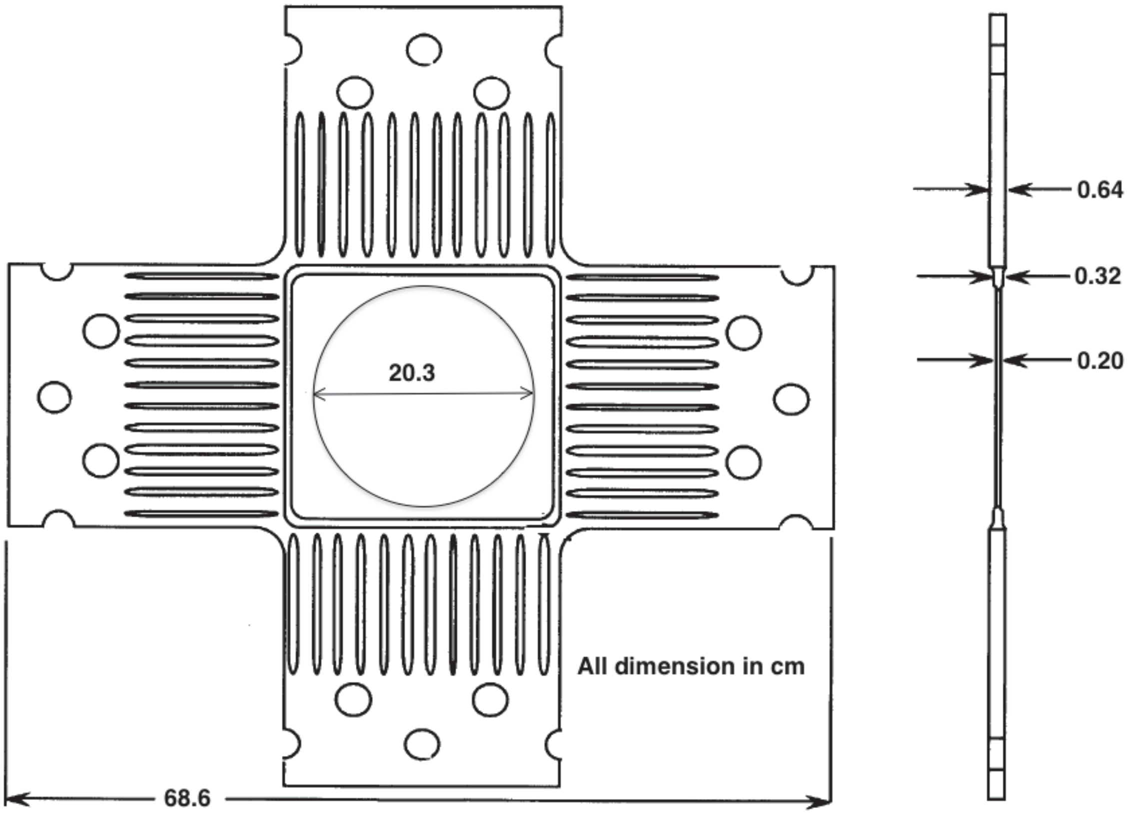

Figure 4. Schematic of the metallic cruciform specimen

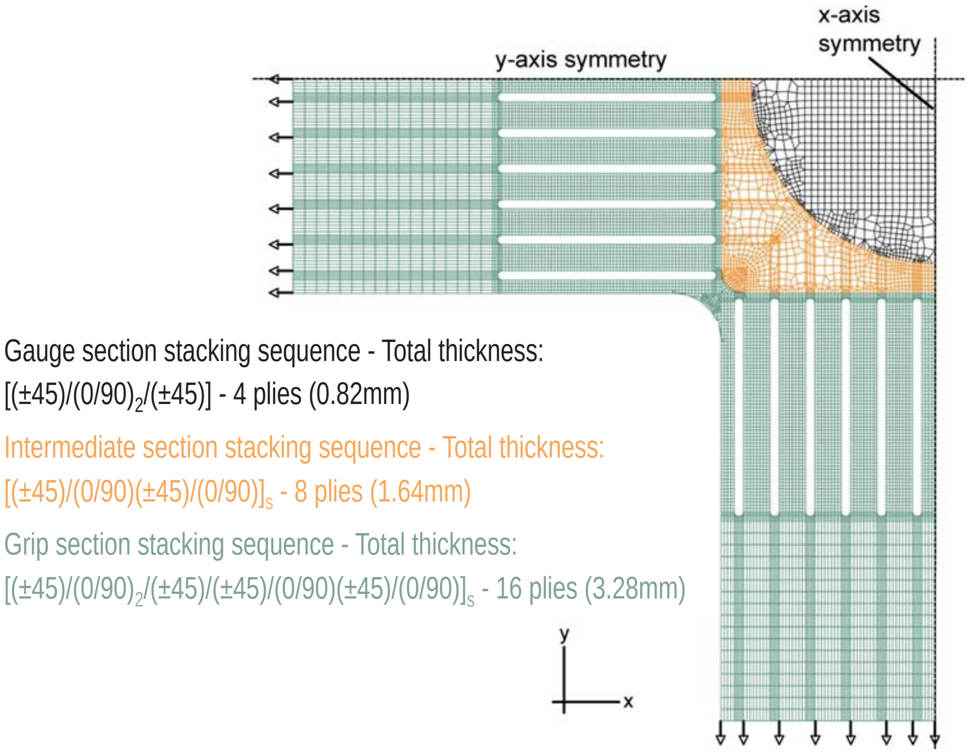

Figure 5. Finite Element Mesh of Cruciform Specimen

TABLE 1. MATERIAL PROPERTIES OF IM7/977-2, 4-HARNESS FABRIC.

E_1=80.7GPa	E_2=74.5GPa	v_{12}=0.036
G_{12}=5.8GPa	G_{13}=5.2GPa	G_{23}=5.2GPa

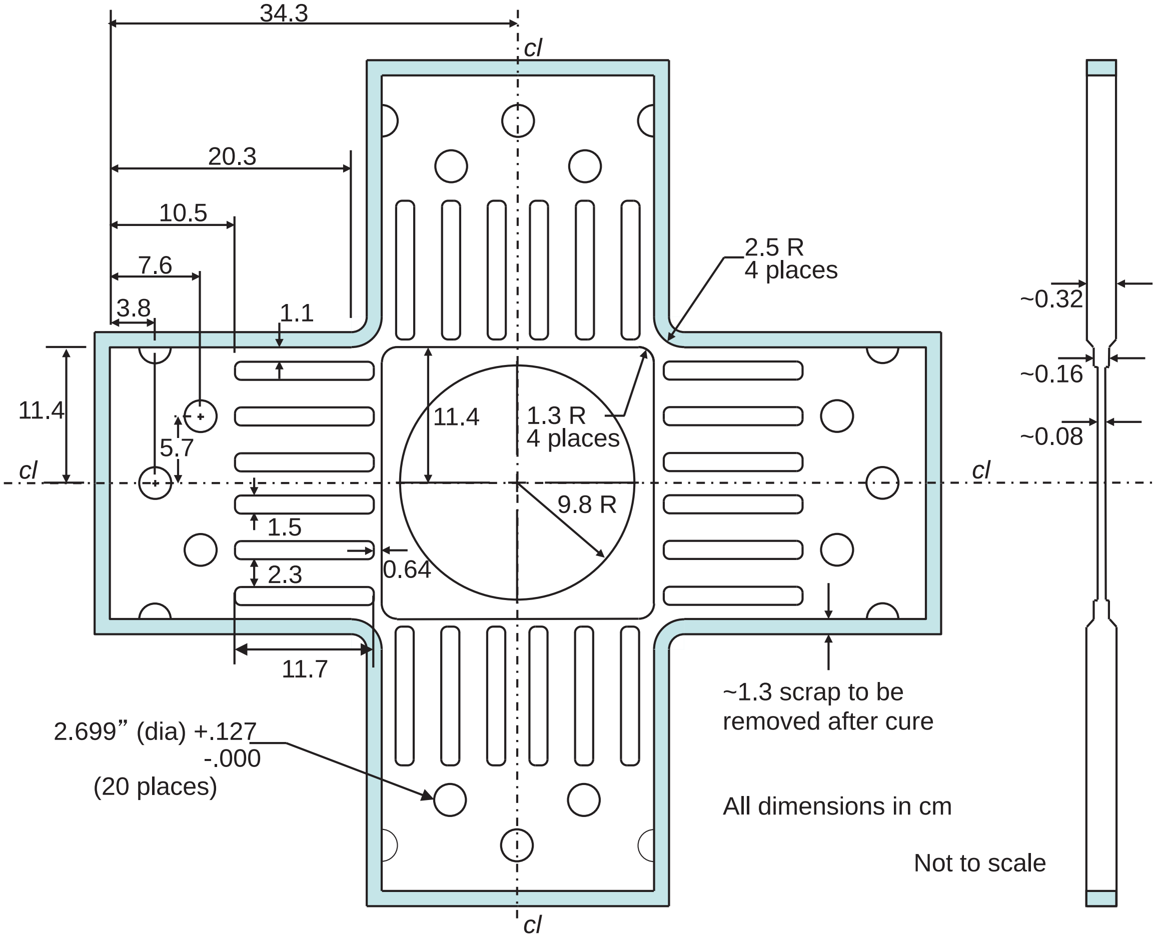

Figure 6. Schematic of the final composite cruciform specimen design

Results from the finite element analysis of the specimen configuration deemed to be acceptable for use are shown in Figure 7. The contour plot in the gage section corresponds to the state of biaxial strain (i.e., 1-direction strain normalized by 2-direction strain referred to as strain ratio), where green corresponds to a pure biaxial state and red and blue correspond to a strain ratio of 1.18 and 0.8, respectively. Computed maximum stress and strain values are also included, in addition to the total reaction forces in the respective direction of the two arms considered in the analysis. The dashed lines correspond to the area of the gage section that was involved in the leak testing. It was found that the strain values in this area would not deviate more than 8 percent from a purely biaxial state.

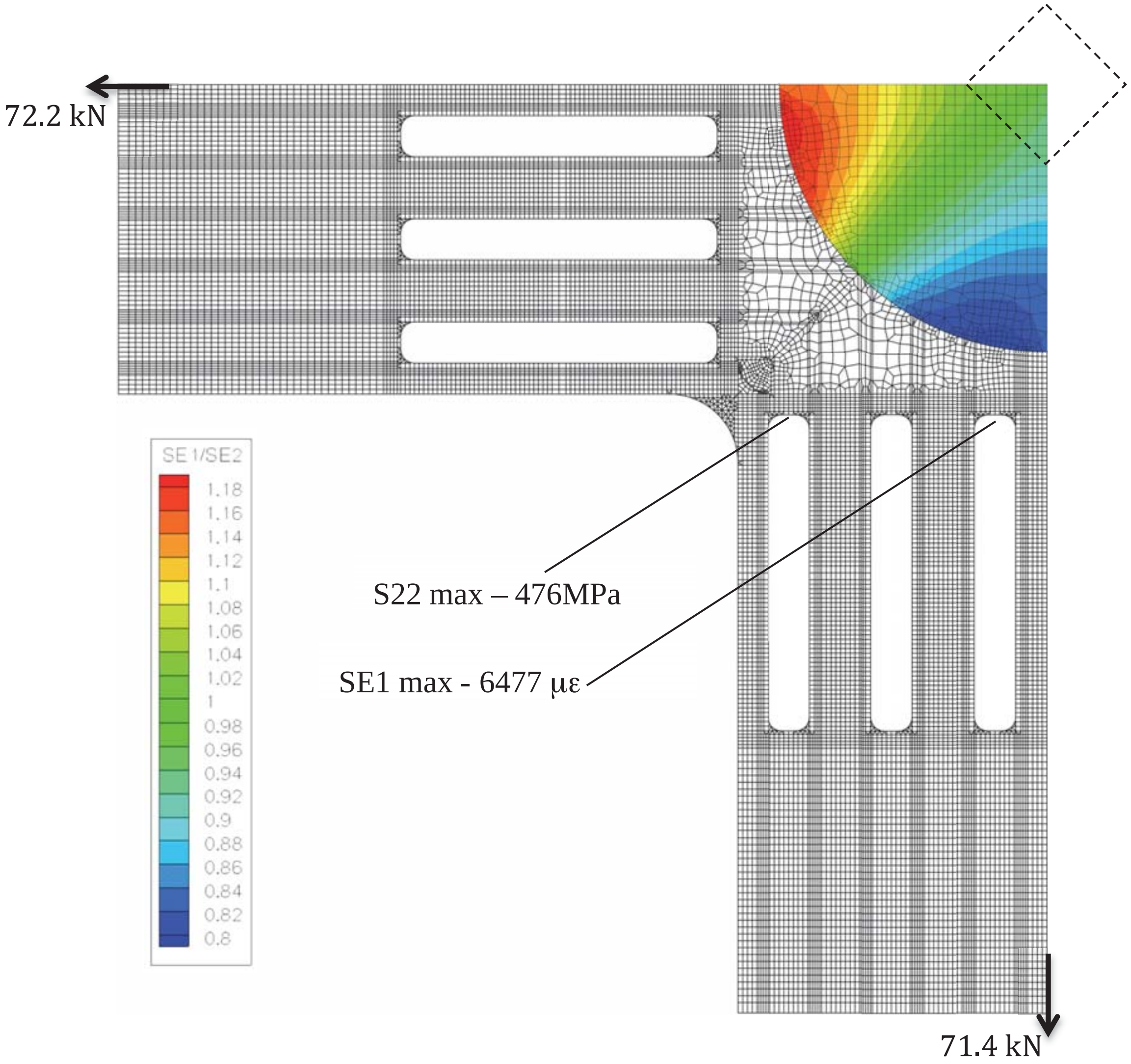

Figure 7. Results from finite element analysis of chosen specimen configuration

The leakage of the material was evaluated by pressurizing a central section of the gage area of the cruciform specimen while it was loaded in the biaxial load frame. The proposed test procedure first involved loading the cruciform specimen until a biaxial state of strain (4000με) was developed in the gage section, with the leakage fixture attached to the central section of the specimen's gage area (58 square cm). A pressure of 172 kPa was then applied to this 7.6-cm square section. An important question to answer was whether or not the pressure loading significantly altered the state of strain in the gage section of the specimen from the intended value of 4000με. During preliminary pressure testing of an uninstalled specimen, a strain of approximately 1900με was recorded on the center gages for a pressure of 172 kPa (gages located on the opposite side of the pressurized side of the specimen referred to here as the outer surface). Consequently, each previous analysis was repeated with a pressure loading of 25 psid applied to the gage section area corresponding to the location of the leakage test fixture. The surface strains in the two global directions were computed. The resulting surface strains on each side of the specimen at the center of the gage section are presented in Figure 8 for the specimen configuration that was chosen for testing. Note that 1-direction strains were used in the results in Figure 8. The 2-direction strain yielded almost identical results. The computed strain distribution when no pressure loading was applied is also included in Figure 8. The comparison highlights that the pressure only perturbs the inner and outer surface strains from their original magnitudes by -2.0 and 4.7 percent, respectively. It was therefore concluded that the pressure application did not adversely affect the strain distribution in the gage section of the specimen at the desired strains. Also for comparison, five experimentally measured strains (average of the 0° and 90° directions on the rosette) are also plotted and correspond to the outer surface strains. The experimental data matched the strains from the finite element analysis extremely well. Details on the biaxial testing of the specimen are given in the following sections.

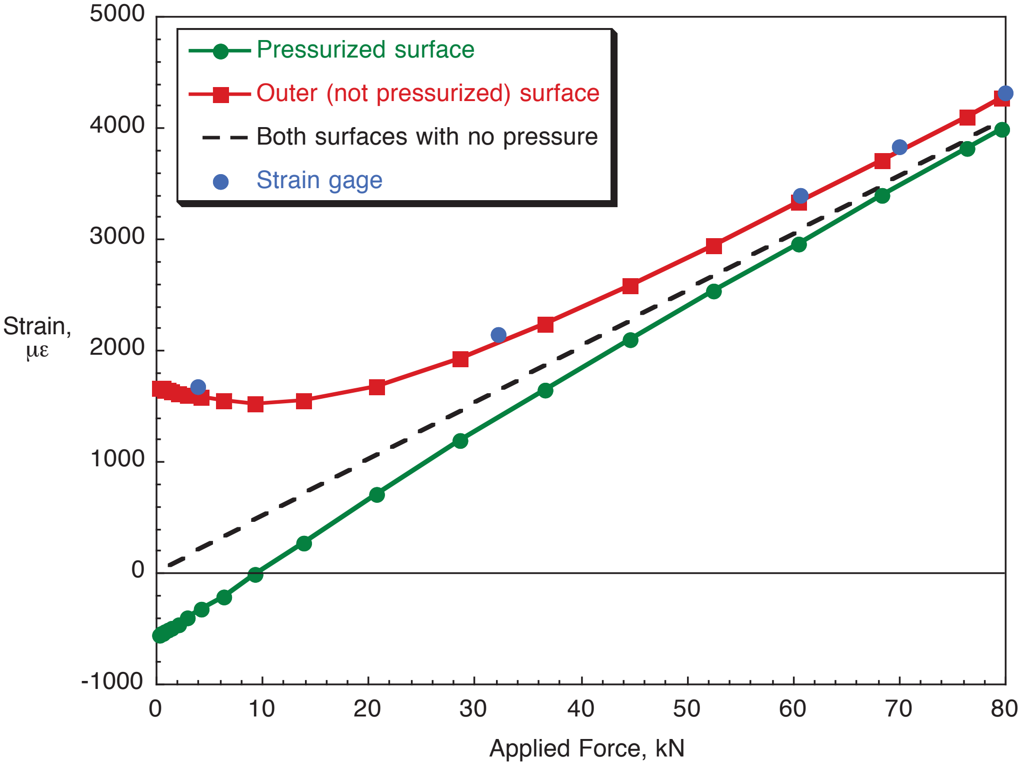

Figure 8. Effect of Pressure on Surface Strains at the Center of the Specimen

TEST SPECIMEN MANUFACTURE AND PREPARATION

After the specimen design was finalized, manufacturing took place at ATK's Composite Structures Center of Excellence in Iuka, MS. Each layer of fabric was cut to the appropriate shape using an automated flat bed pattern cutter and an ultrasonic knife. To match the appropriate contours on the top and bottom of the specimen, the portions of the fabric that were removed from the interior sections from each layer were returned to their original position for autoclave curing. However, these pieces were separated from the specimen with a PTFE film during the cure so they could be removed afterwards. The use of this technique resulted in a high quality specimen without the need for specialized tooling.

Following the cure, the specimen was sent to NASA Marshall Space Flight Center for final machining. The machining included removing approximately 1.3 cm of material from the edges and milling the slots and holes. The procedure for installing the specimen in the biaxial test machine requires that the specimen be pin loaded through the center holes of each loading arm to achieve an acceptable alignment. The standard alignment procedure for a metallic specimen uses a 22 kN load for this step. Therefore, a bearing test was performed that showed that a bearing failure would initiate prior to reaching the alignment load. Consequently, tabs were required to prevent damage to the specimen during this initial alignment step. Tabs made from 2.29-mm aluminum sheet were bonded onto each side of the loading arms (Figure 9). To increase the friction between the grips and the specimen tabs, a P150 grit silicon-carbide open-mesh sheet was placed at each interface as shown in Figure 9.

The rosettes were bonded near the corners and were opposite the rosettes on the back surface (Figure 9). A two-gage rosette was bonded to the center on one side to measure the biaxial strains in the area of interest. The area outside this rosette was masked off prior to installing the strain gages to keep it free from adhesive and gage coating for leak testing.

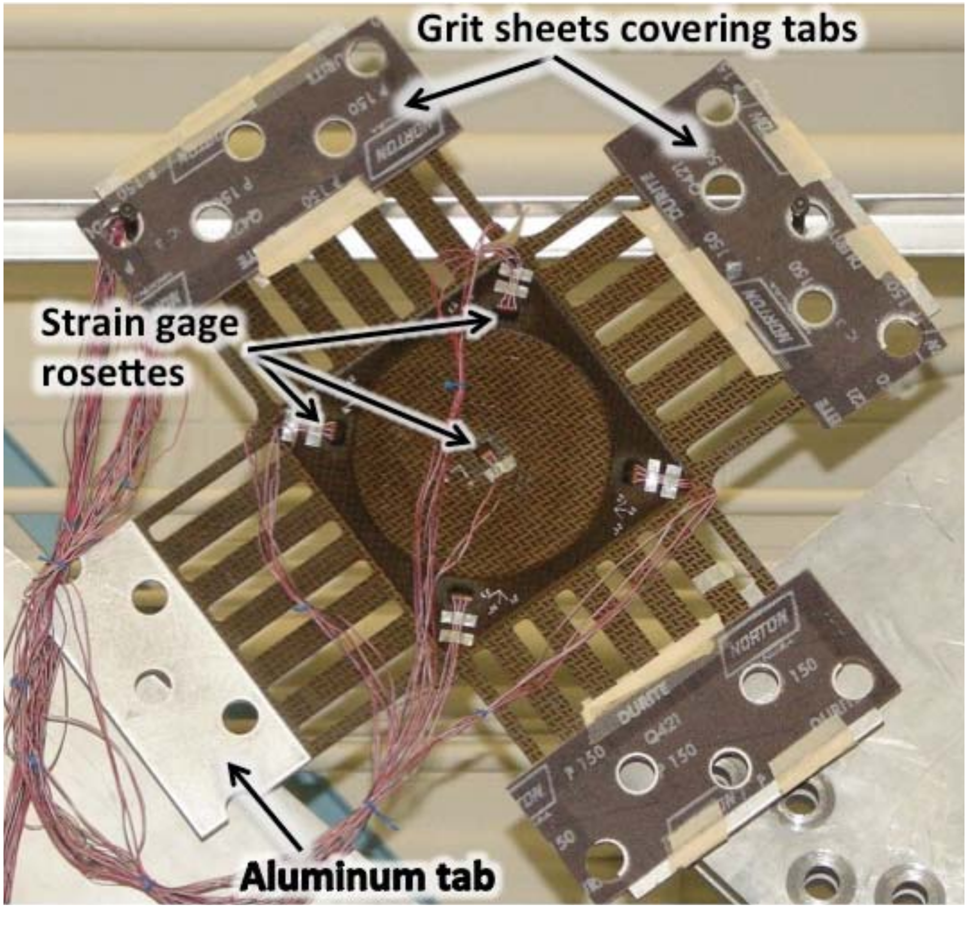

Figure 9. Biaxial Specimen Showing Aluminum Tabs and Strain Gages

MECHANICAL TESTING AND RESULTS

Prior to leak testing, a series of preliminary tests was run to evaluate the overall response of the specimen to loading and pressure. After the specimen was installed with an acceptable alignment (refer to Biaxial Test Machine Procedures), the biaxial strain ratio at the center and the ratio of forces between the loading axes were initially investigated. The strains and actuator forces were recorded while the actuators were displaced to increase the average biaxial strains between 3000 and 4500$\mu\varepsilon$. The ratio of forces between the two loading axes averaged 1.07 to 1 and the ratio of strains at the center (biaxial strain ratio) averaged 1.14 to 1. Both ratios remained relatively constant (<2% variation) over this strain range. This biaxial strain ratio was considered acceptable for conducting the leak tests.

Next, the effect of the pressure differential on the central strain gages was investigated. A previous pressure test of an uninstalled specimen showed a pressure differential of 172 kPa resulted in center strains of approximately 1900$\mu\varepsilon$. The aluminum leak fixture was attached to the specimen while loaded (Figure 10). An aluminum bar was attached to each half of the leak fixture to provide the clamping force. Bolts at either end of the bars, near the specimen's outer contour radius, were tightened to provide a uniform clamping pressure. The strains at the center gages were recorded as a function of applied force and pressure. A plot of the average center strain for four different pressure differentials is shown in Figure 11 as a function of the average force of the four actuators. With no pressure applied, there is a linear relationship between the actuator force and center strain as expected. However, when the specimen is lightly loaded, the applied pressure does increase the center strains significantly. For the lowest loads, the applied pressure increased the center strains by more than eight times. However, at an initial strain of 3180$\mu\varepsilon$, the 172 kPa of pressure difference increased the average center strain by 6.7 percent relative to the unpressurized specimen. Similarly for an initial strain of 4150$\mu\varepsilon$, the average center strain increased by only 4.0 percent. This increase is nearly identical to what was predicted by the analysis in the previous section. Consequently, for the strains of interest, the pressure did not appear to significantly affect the strain or the strain ratios based on these experiments and the finite element analysis.

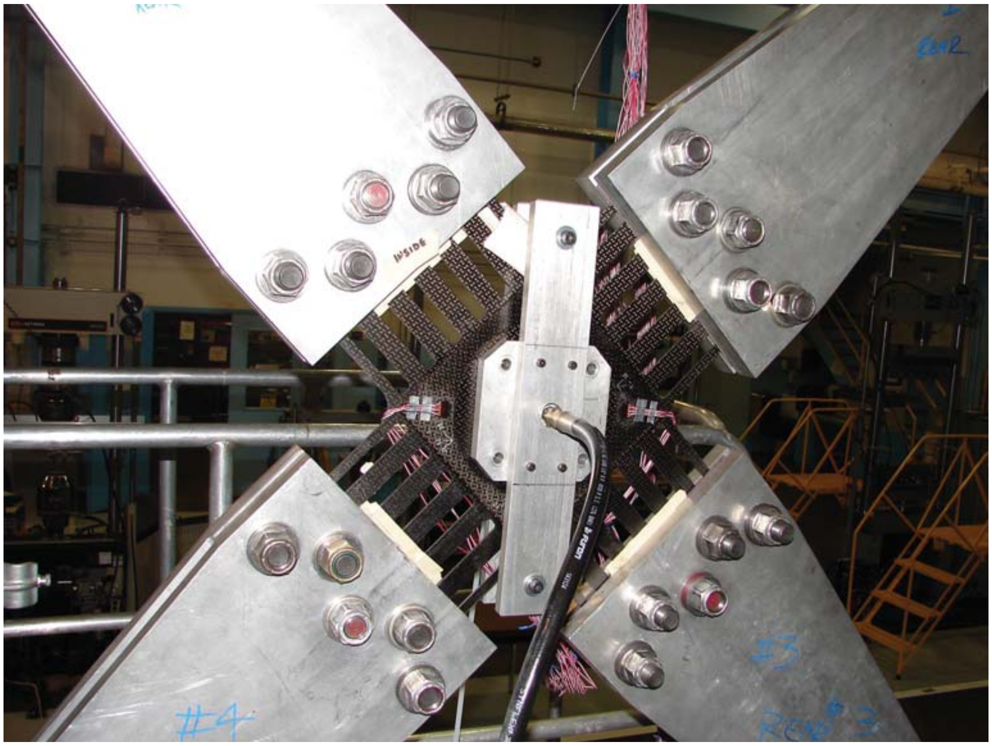

Figure 10. Specimen in biaxial load frame with leak measurement fixture attached

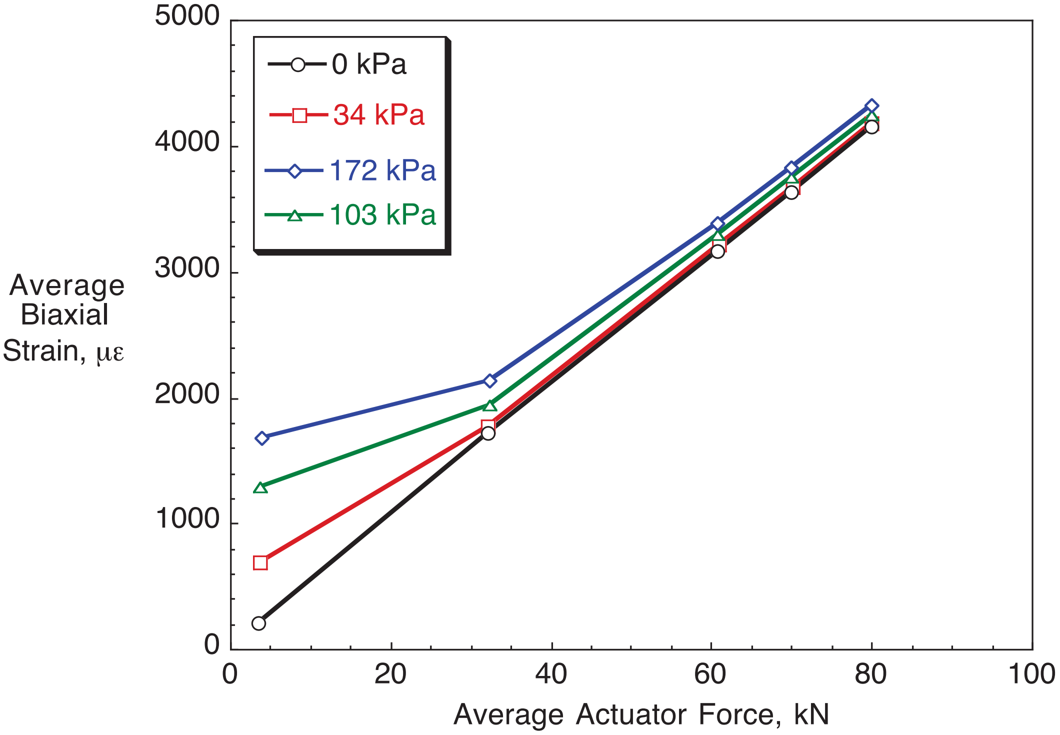

Figure 11. Average Center Strain versus Average Actuator Force for Various Pressures

A series of leak tests was performed to determine the leak response as a function of biaxial strain and pressure differential. The leak measurement fixture was attached to the specimen after the specimen was under load. The desired strain levels were set by simultaneously increasing the displacement of all four actuators while monitoring the strains of the center gages. After the desired strain level was achieved, flow measurements were made at pressure differences of 34, 103, and 172 kPa. The leak measurements were performed at initial average center strains (prior to pressure application) of 200με, 1830με, 3170με, 3870με, and 4150με. As previously mentioned and shown in Figure 11, the center gage strains during the leak measurement tests were above the initial value due to the out-of-plane deformations caused by the pressurization. For all 15 leak tests, no leakage was measured within the accuracy of the leak measurement equipment (approximately 1-2 sccm). While the measured strains were higher than nominal due to the pressurization, the pressurized surface had slightly lower strains and would be less likely to leak.

CONCLUDING REMARKS

A new composite cruciform specimen was designed for leak testing based on an existing metallic design. The specimen featured a large flat region of relatively uniform biaxial loading where leak testing could be performed. A large test section was desired to accommodate future tests containing impact damage. The composite cruciform specimen was demonstrated to meet the test requirements under biaxial loading. Due to the 4-ply test section and slotted loading arms, the specimen was flexible but could be installed without damage. Tabbing material was required in the grip regions to accommodate the stresses caused by the pin-loading alignment procedure. The design was loaded to an average biaxial strain of approximately 4500με without apparent damage. A biaxial strain ratio of 1.14:1 was achieved in the tests rather than the desired 1:1. This was primarily attributed to slight differences in the forces required on each axis to maintain the central position of the specimen in the test frame.

Each half of the leak measure fixture was clamped on opposite sides of the specimen to apply the pressure differential. This pressure did result in an out-of-plane deformation that resulted in differential strains on opposite sides of the test area. This difference was greatly reduced when tensile loads were applied to the specimen. To limit this deflection, a rigid screen or porous material could be placed on the unpressurized side. The leak rates were measured using a system that used flow meters and a pressure controller. Although this system did not have the capability to measure extremely small flow rates (< 1-2 sccm), no leakage was measured through the specimen for biaxial strains up to 4150με. If greater measurement sensitivity to leakage is required, a more sensitive leak measurement system could be added to this test configuration. Other materials that are more susceptible to microcracking may leak at these strains. The IM7/977-2 material has been previously shown to be more resistant to microcracking and leaking.

An alternative biaxial test method for determining leakage relies on a pressurized disk to obtain both the biaxial strain field and the pressure differential to drive the leakage while visually monitoring the surface using a leak detection fluid. Relative to the disk method, the cruciform specimen with a separate leak measurement system allows for independent control of the biaxial strain field and pressure differential as well as a quantitative measurement of the leakage rate. This method also offers significantly safety advantages since much lower pressures are used in smaller volumes relative to the high pressures required for the disk method. However, the cruciform test specimens are expensive to produce and require a specialized multi-axial test machine. An additional advantage is that the cruciform specimens could be subjected to cyclic biaxial loading in order to create matrix cracking since hydraulic actuators are used.

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