JOURNAL OF SPACECRAFT AND ROCKETS
Vol. 42, No. 2, March–April 2005

Engineering Notes

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Experimental Cryogenic Gas Leakage Through Damaged Composite Laminates for Propellant Tank Application

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Nomenclature

f = temperature correction factor
T_{LN_2} = absolute boiling temperature of nitrogen
T_{RT} = absolute room temperature

Introduction

CARBON-FIBER-REINFORCED plastics (CFRP) are widely used as structural components and are regarded as the major candidates for reducing structural weight of future reusable launch vehicles (RLV). Application of CFRP laminates to cryogenic propellant tanks is one of the most desirable but challenging technologies for achieving drastic weight reduction of RLV. Recent basic studies on the feasibility of composite liquid propellant tanks indicated that matrix crack onset and its accumulation are inevitable when conventional high-performance composites are applied to cryogenic tanks and multiple-ply matrix cracks may induce detrimental propellant leakage.[1]

Although several investigations have attempted assessment of the feasibility of composite propellant tanks for space vehicles using subscaled structures or prototypes,[2,3] basic research on leakage mechanisms in composite structures are rare to the authors' knowledge. Kumazawa et al.,[4] Aoki et al.,[5] and Kumazawa et al.[6] investigated helium leakage through cross-ply laminates with matrix cracks in all constituent plies at room temperature (RT) and clarified the effect of biaxial loadings on fuel leakage, claiming that the damage-induced permeability may exceed tolerable levels for propellant tanks. A simple semianalytical model based on leakage conductance at crack intersections, which is related to the crack opening displacements of the adjoining plies, was developed and effects of mechanical and thermal loads, crack density, and ambient temperature on gas leakage were analytically investigated.[5,6]

In this study, experimental characterization of gas leakage under cryogenic conditions through CFRP laminates is highlighted in reference to the analytical investigations.[5,6] Tensile loadings are applied to the CFRP tubular specimens utilizing the cryogenic loading system developed previously[1] for leak flux measurement through damaged laminates with a helium leak detector. A comparison of gas leakage at RT and liquid nitrogen (LN_2) temperature under identical damage is presented.

Experimental Procedures

Materials and Specimens

The material system used in this study was IM600/#133, an intermediate-modulus carbon fiber and toughened epoxy system. To examine the through-the-thickness permeability of composite laminates, laminated tubular specimens were prepared in place of the cruciform specimens that were utilized in the research of Kumazawa et al.,[4] Aoki et al.,[5] and Kumazawa et al.[6] All tubular specimens were 200 mm long with 50-mm CFRP end-tabs, leaving a 100-mm gauge section, and the inner radius was 30 mm. Four types of specimens with embedded or surface 90-deg layers were prepared; $[45/-45/90]_s$, $[45/-45/90_2]_s$, $[90_2/-45/45]_s$, and $[90_2/0/90_2]_s$. Nominal thickness of each ply was 0.15 mm.

Test Flow

The major objectives of this study are to measure the gas permeability of cracked laminates under cryogenic conditions and to compare the gas leakage of laminates under identical damage at RT (22°C) and LN_2 temperature (−196°C). The test flow was as follows:

Step 1. Helium gas permeability is measured under LN_2 conditions during application of static tensile loads up to the level at which high-level leakage is observed, followed by leak measurements under decreasing load steps.

Step 2. Helium gas permeability is measured at RT with limited load levels so that further cracks in tubular specimens are not introduced.

According to this test flow, helium leak flux of the identical damaged specimens at room and LN_2 temperature could be measured as a function of applied mechanical load.

For the detection of matrix cracks in the tubular specimens, ultrasonic inspection with high-frequency probes (25 MHz) was conducted (commission to GNES Co., Ltd.) before and after permeability measurement. Matrix cracks have been successfully identified by use of this ultrasonic technique in cruciform[1,4–6] and tubular[7] specimens.

Cryogenic Permeation Test (Step 1)

A tensile loading fixture made of low-thermal-expansion alloy with flow path was bonded to the tubular specimen, whose outer surface was wrapped with a polyester sheet in a skirt-like shape. Next, the specimen was attached to the cryogenic loading system.

Received 1 February 2004; accepted for publication 8 October 2004.

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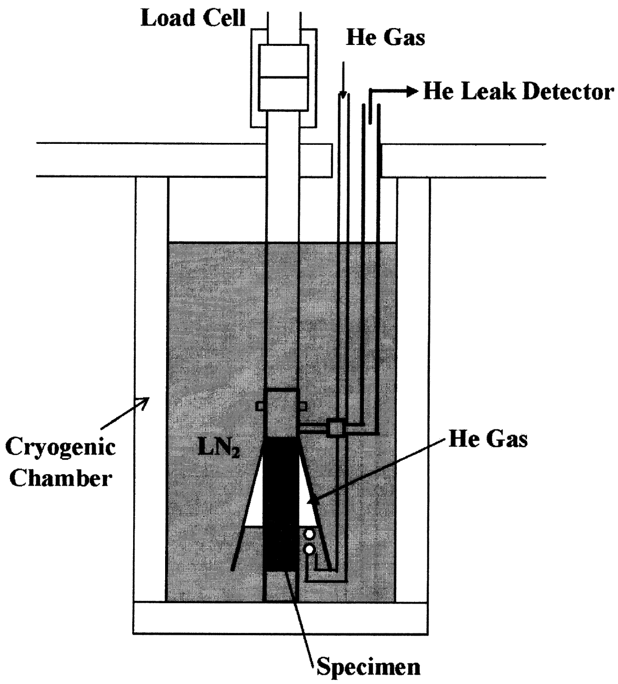

Fig. 1 Schematic of cryogenic permeation test.

The inner portion of the tubular specimen was connected to the helium leak detector with a vacuum pump and was subjected to vacuum conditions. After liquid nitrogen was introduced into the cryogenic loading system, helium gas was supplied from the outer surface of gauge sections, and thus helium permeation tests were performed under a pressure difference of 1 atm (about 100 kPa). Note that the head pressure due to LN_2 was less than 5 kPa and thus was negligible. The schematics of these permeation tests are shown in Fig. 1.

After specimens were left without loading for 30 min, static tensile loading was applied to the tubular specimen with crosshead speed 0.5 mm/min. Once substantial leakage was observed, the specimen was unloaded with decreasing steps. The relationship between applied mechanical loadings and gas leakage was obtained.

Room Temperature Permeation Test (Step 2)

A tubular specimen identical to that used in the cryogenic test was installed in the loading system at RT and connected to the helium leak detector. The differential helium pressure was set to be 1 atm. Static tensile loading was applied to the specimen with cross-head speed 0.5 mm/min up to the mechanical strain range of 0.30–0.40%, which was far below the threshold strains[7] of matrix crack initiation in 90-deg plies under RT, in order not to induce further cracking. Thus, the relationship between the applied load and gas leakage was obtained by holding loads at specific strain levels.

Experimental Results

The relationship between applied mechanical loadings and helium leak flux of a [45/−45/90]s specimen in cryogenic circumstances is shown in Fig. 2a. When applied strains reached about 0.7%, a sharp increase of leak flux was observed. Subsequently, the load was decreased and helium permeability decreased in conjunction with load decrease. The value of leak flux indicated no less than 10^{-6} Pa m^3/s even without tensile loadings, which provoked the inference that matrix cracks accumulated in all constituent plies and damage-induced leakage took place. Another example of a [90_2/0/90_2] laminate is shown in Fig. 2b. All other specimens showed the same tendency to sudden leakage and leak flux changes.

The values of applied mechanical strains at which sharp increases in leakage were observed at LN_2 temperature are summarized in Table 1. This indicates that laminates with thinner 90-deg layers had more capacity for leakage prevention, whereas

Table 1 Applied mechanical strains at onset of substantial leakage in cryogenic test

Layup	Strain %
[45/−45/90]s	0.69
[45/−45/90_2]s	0.46
[90_2/−45/45]s	0.53
[90_2/0/90_2]s	0.55

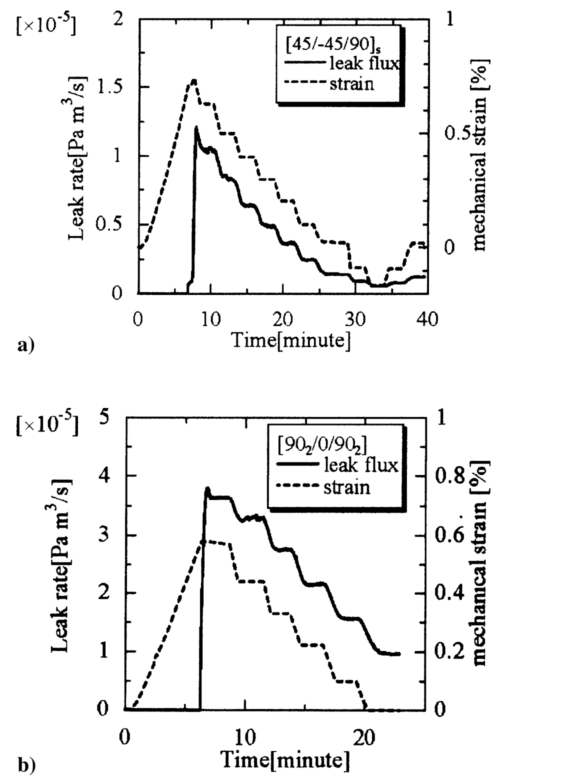

Fig. 2 Results of cryogenic permeation test in conjunction with tensile loadings plotting leak rates and applied loadings as a function of time: a) [45/−45/90]s and b) [90_2/0/90_2]s.

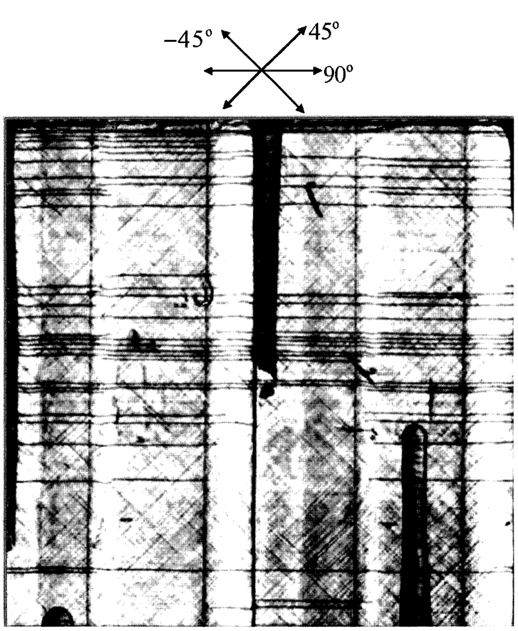

Fig. 3 Deployed ultrasonic image of a cracked [45/−45/90]s tubular specimen after step 2.

high-level leakage could be observed in $[90_2/0/90_2]$ specimens (laminates containing a 0-deg layer) at a strain level similar to that at $[90_2/-45/45]$s.

Helium leak flux of the same specimens used in the cryogenic permeation tests was measured at RT with step loadings. Similar high-level leakage was observed for all of the tubular specimens that exhibited substantial leakage during cryogenic permeation tests. The leak rates had an apparent correlation with the applied mechanical loadings in cryogenic tests. The ultrasonic image of this specimen after the RT step indicated that numerous matrix cracks had accumulated not only in 90-deg layers but also in other ±45-deg layers, as shown in Fig. 3. This leads to the conclusion that the connections of these matrix cracks through the thickness direction induced high-level helium leakage.

Discussion

Measured leakage data at room and LN_2 temperature obtained in this study include both "thermal" and "mechanical" effects. The thermal effect represents temperature-dependent molecular activity, whereas the mechanical effect comprises the changes of damage scale due to both the mechanical and residual thermal strains. Thus, the leak rates at room and LN_2 temperature utilizing the same specimens with the identical damage states (numbers and sizes of cracks) are expected to differ because of the combined effects. The

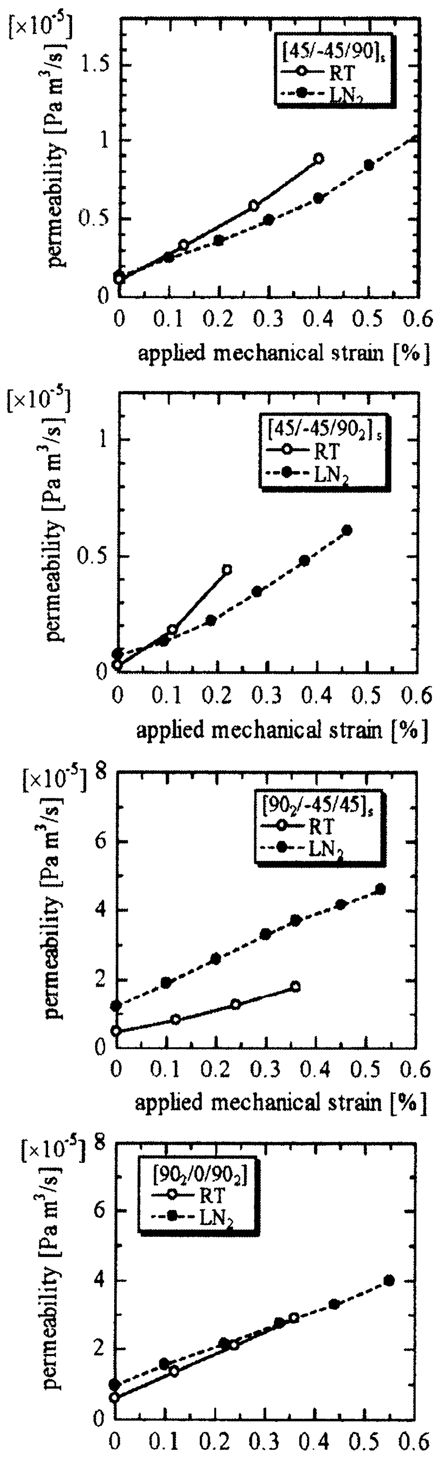

Fig. 4 Comparison of leak rates of cracked tubular specimens at LN_2 temperature and RT: raw data.

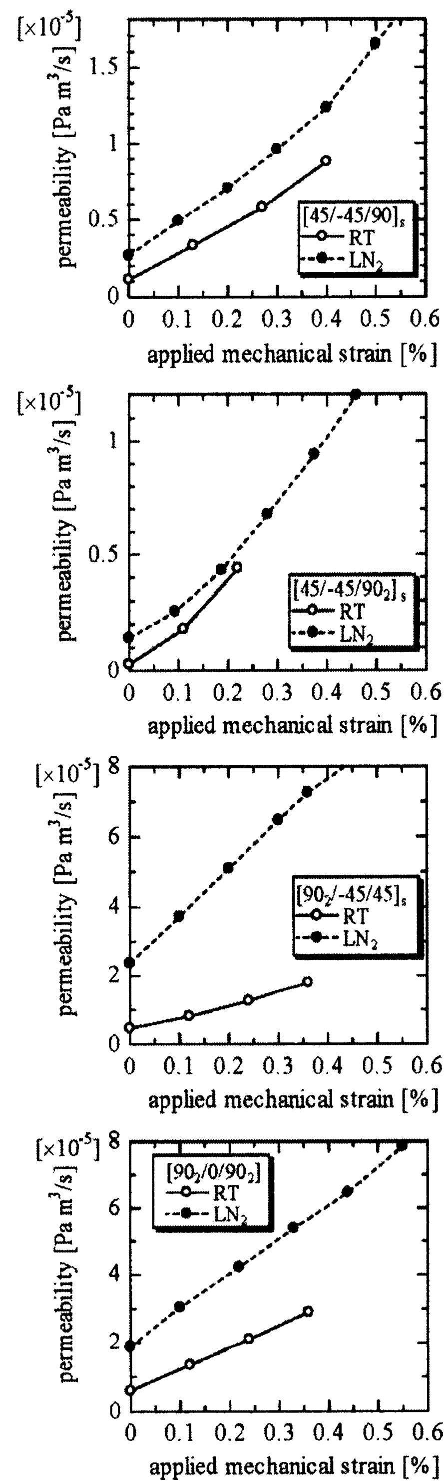

Fig. 5 Comparison of leak rates of cracked tubular specimens at LN_2 temperature and RT: compensated data.

comparison of the leaks flux (raw data) at both temperatures is shown in Fig. 4. The observed helium leakage turned out to differ only slightly between the two temperatures.

In order to separate the thermal and mechanical effects from the obtained leak rates, temperature compensation is carried out. Theoretically, leak mass flux through the orifice is in proportion to the square root of the absolute gas temperature. Thus, the measured leak rates under LN_2 conditions are multiplied by the temperature correction factor f, defined as

$$f = \sqrt{T_{\mathrm{RT}} / T_{\mathrm{LN_2}}} = \sqrt{295/77} \approx 1.96 \tag{1}$$

With this compensation, the difference due to the effect of crack opening displacements under room and LN_2 temperature is extracted.

The compensated comparison is shown in Fig. 5 for all the four layups. These results exhibit higher leak rates under LN_2 conditions than under RT for all cases, indicating that gas leakage increases under cryogenic conditions because of the increase of the leak path size due to the residual thermal strains. This tendency is well compatible with the analytical prediction presented by Aoki et al.[5] and Kumazawa et al.[6] These experimental findings also verify that the thermal effect of cryogenic conditions contributes to a suppressive role on the damage-induced gas leakage due to low molecular kinetics, whereas the mechanical effect provokes the leakage increase. Further research on the identification of leak path cross-sectional areas and quantitative correlation between experiments and analytical predictions under cryogenic conditions should be carried out for the feasibility assessment of composite cryogenic propellant tanks.

Conclusions

Leakage characteristics through microcracked laminates under cryogenic conditions were experimentally investigated using helium gas as the fundamental research for the development of composite cryogenic propellant tanks. High-level leakage could be observed for all of the specimens used herein under LN_2 conditions in conjunction with tensile loadings. Ultrasonic inspection indicated that matrix cracks exist in all of the constituent plies, forming leakage paths and causing extensive leakage at LN_2 temperature and RT. Utilizing the obtained leakage results, comparison of leak rates at LN_2 temperature and RT was performed. The results indicated that leakage levels differ only slightly between two temperatures. However, with temperature compensation, the mechanical effect of cryogenic conditions turned out to have significant effect on increasing gas leakage, whereas the thermal effect due to low temperature reduces the leakage because of the low molecular kinetic energy.

Acknowledgment

The authors acknowledge Yoichi Hayashi from Tokyo Business Service for his invaluable contributions to this test program.

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Associate Editor