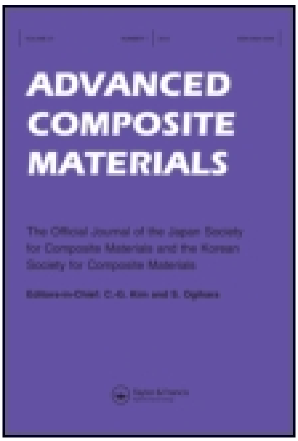


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# Cryogenic mechanical properties of CF/polymer composites for tanks of reusable rockets

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# Cryogenic mechanical properties of CF / polymer composites for tanks of reusable rockets

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**Abstract**—Cryogenic properties of different types of CFRPs are experimentally evaluated to survey the basic applicability of different material systems to the cryogenic propellant tanks for future reusable launch vehicles. Temperature dependent material constants, tensile strength and interlaminar fracture toughness are experimentally obtained, together with detailed observations of matrix cracks and delaminations. Up to about 20% reduction in cryogenic static tensile strength is observed for most of the material systems tested. The damage initiation stresses also decreased under cryogenic conditions. The results indicate that matrix cracks may be one of the major critical issues when current material systems are applied to cryogenic propellant tanks. Numerical predictions of the delaminations and matrix cracks are conducted to theoretically support the experimental consequences.

*Keywords*: Mechanical properties; cryogenics; propellant tank; matrix crack; delamination; interlaminar fracture toughness.

## 1. INTRODUCTION

The wide use of composite materials is the major technical challenge for effectively reducing the structural weight of the future reusable launch vehicles. The cryogenic propellant tanks are the dominating structural components of the vehicle structure and thus the application of carbon fiber reinforced plastics (CFRP) to these components is one of the most promising technologies for realizing the aimed weight reduction. In this study, the basic cryogenic mechanical characteristics of CFRP laminates are experimentally and analytically evaluated to discuss their applicability to the propellant tanks.

**Table 1.**
CFRP material systems

| Material designation | Supplier | Fiber | Matrix resin |
|---|---|---|---|
| *Aa* | Toho Rayon | IM600 | 133 (180°C-cure epoxy) |
| *Ab* | | HTA | 112 (120°C-cure epoxy) |
| *Ad* | | HTA | 332 (bismaleimide) |
| *Ae* | | IM600 | PEEK |
| *Ba* | Mitsubishi Rayon | MR50K | 982 (180°C-cure epoxy) |
| *Bb* | | MR50K | 154 (120°C-cure epoxy) |
| *Ca* | Mitsubishi Chemical | IM7 | 977-2 |



## 2. MATERIALS AND MATERIAL PROPERTIES

The materials are based on different types of epoxy matrices, bismaleimide matrix and PEEK (Table 1). Type *Aa*, which is the combination of intermediate modulus fiber and toughened epoxy, is herein set to be the base material and has been investigated in detail.

The existing data of cryogenic mechanical properties are often provided for specific discrete temperatures such as $LN_2$(−196°C) or LHe(−269°C) conditions [1]. In this respect, it is worthwhile to present the temperature dependent mechanical properties as continuous functions of temperature. The temperature dependent anisotropic elastic constants and thermal expansion coefficients for material *Aa* are obtained based on the experimental measurements and past references (Fig. 1). They are used later in the analytical predictions of delamination propagation and matrix crack onset. It should be noted that when wide range of thermal loading up from room temperature down to cryogenic condition is of interest, temperature dependent material properties must be taken into account in the analyses.

## 3. ANALYSIS

The quasi-isotropic laminate with the single stacking sequence of $(45/0/-45/90)_{2S}$ is employed here for analytical predictions to be compared with the following experiments. The energy release rates are calculated for delamination propagating at each ply interface to identify the location of possible delamination onset. The calculation for free-edge delamination is based on the simplified method to calculate the saturated value [2]. The analytical results predicted that the inner $-45/90$ ply interface is the most susceptible location for delamination to take place. This coincides with the experimental observations that the delamination always appeared at the same interface.

The analytical method employed herein to calculate the energy release rates associated with the matrix crack onset is based on the scheme proposed by Park and McManus [3]. The central $90_2$ layers are assumed to be the location for initial matrix

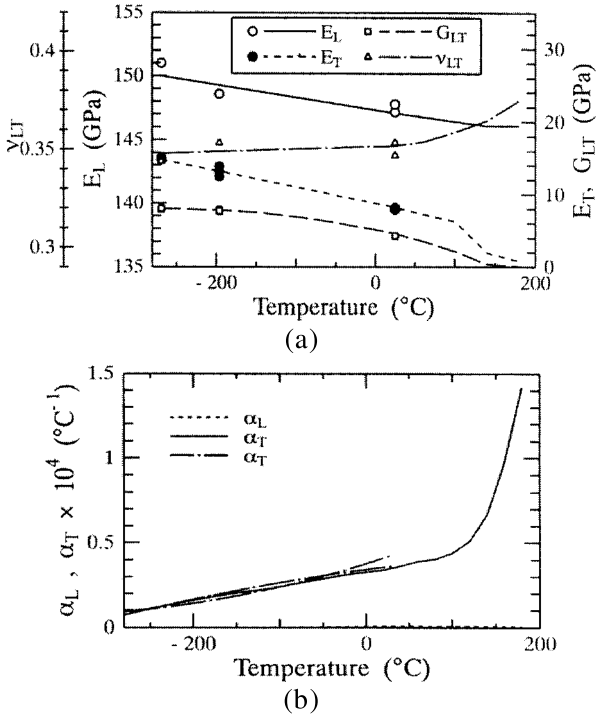


**Figure 1.** Temperature dependent material properties of intermediate modulus CF/180°C-cure touhened epoxy unidirectional composite (Type *Aa*, IM600/133). (a) Elastic moduli. (b) Thermal expansion coefficients.

cracks to appear, as the thicker plies are usually more apt to damage initiations. The analytical results suggest that, for the quasi-isotropic laminates considered herein, the mechanical tensile loads at matrix crack onset are drastically lowered under cryogenic condition, compared to those under room temperature. This suggests that the appearance of matrix cracks could be a critical issue under cryogenic conditions. The present energy release rate calculation for matrix crack onset is applied later to interpret the experimental results from the fracture toughness point of view.

## 4. EXPERIMENTAL

### *4.1. Experimental setup*

Static tensile tests are conducted using specimens of nominal thickness and width of 2.2 mm and 15 mm, respectively. The loading system utilizing 4-rods turret type tension introduction device has been developed and combined with the surrounding double-walled cryogenic chamber. This system is installed in the Instron 4505 Testing Machine (Fig. 2). This device facilitates successive loading of up to four specimens under single setup of the cryogenic environment so as to spare the cryogenic mediums and setup time. The tests were carried out in $LN_2$ and

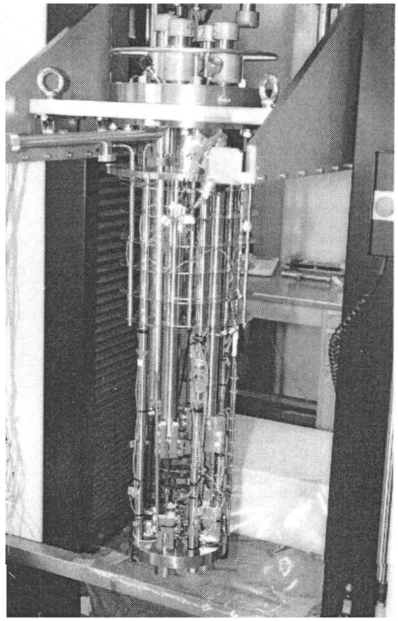

**Figure 2.** Loading apparatus for cryogenic test.



LHe environment to simulate the cryogenic propellant conditions, and at room temperature. The identical setup is also used in the DCB fracture toughness measurements.

### *4.2. Tensile strength and associated damage observation*

The tensile strengths of $(45/0/-45/90)_{2S}$ 16 ply, quasi-isotropic laminates of different material systems are obtained. Three specimens are tested for each temperature condition and averaged for later use. The example test results are shown in Fig. 3 for type *Ae* material system (IM600/PEEK). The tensile strengths for all material systems are summarized in Fig. 4. All of the material systems except type *Ad* exhibit strength reduction of up to about 20% at cryogenic conditions compared to those at room temperature.

Matrix crack onset and delamination propagation are investigated based on the acoustic emission (AE) measurements and direct visual observation. The typical stress–strain curves and associated AE counts are shown in Fig. 5 in which the indefinite correlation between the AE counts and matrix crack onset is seen under LHe condition. Based on this fact, matrix cracks are visually examined for specific specimens during the cryogenic test by halting the loading and removing the specimen from the experimental setup. The initial matrix cracks were observed in the central $90_2$ plies, as predicted, and additional cracks in the adjacent −45 ply were observed at higher load levels in some specimens. The delaminations were found at inner −45/90 interface as predicted by the energy release rate analysis.

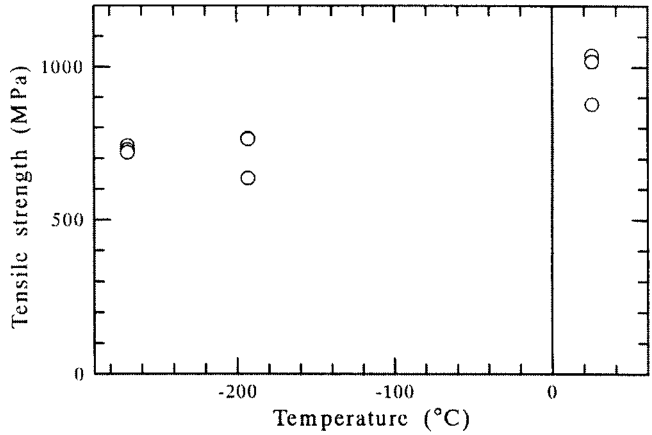


**Figure 3.** Temperature effect on tensile strength for the quasi-isotropic laminates of type *Ae* material system (IM600/PEEK).

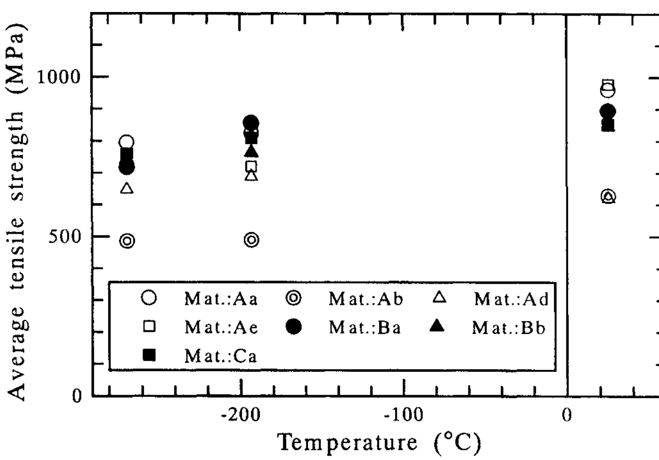


**Figure 4.** Temperature effect on tensile strength for the quasi-isotropic laminates of different material systems.

The thermal effect on the onset of matrix cracks, delamination propagation and static strengths of the laminate is summarized in Fig. 6. For each material type and given temperature condition, all three specimens used for static failure tests are subjected to the damage observations. The temperature dependence of the damage initiations for type *Aa* base material system is shown in Fig. 7. The matrix cracks tend to take place at drastically lower mechanical load under cryogenic environment, which coincides with the numerical predictions. This poses the possibility of fuel leakage through the chain of these matrix cracks. Though it is important to look into the damage accumulation process in detail to investigate the effect of cryogenic environment, this was not performed in the present experiments.

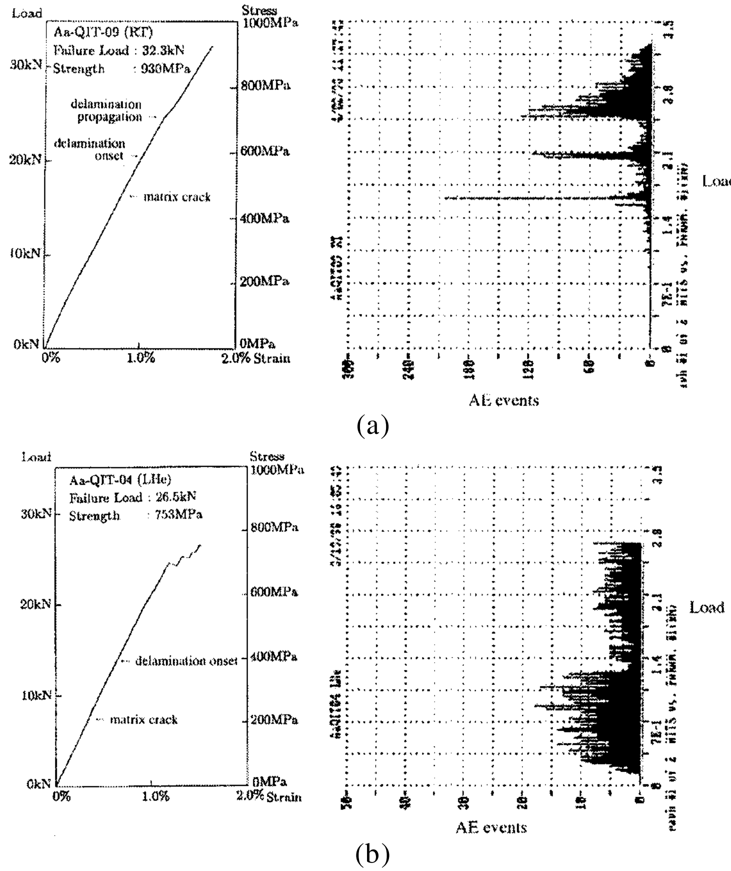


**Figure 5.** Tensile stress–strain curves and AE counts for quasi-isotropic laminates of type *Aa* material system (IM600/133). (a) Room temperature. (b) LHe temperature.



### 4.3. Interlaminar fracture toughness

In order to look into the detailed characteristics of delamination and matrix crack behaviors, it is essential to obtain the interlaminar fracture toughness under cryogenic conditions. DCB specimens of thickness 4.5 mm and width 12.7 mm were used. The interlaminar fracture toughnesses in terms of energy release rate under room and cryogenic temperatures are plotted in Fig. 8. Each plot represents the average propagation value over the full measured crack length range of each DCB specimen.

The fracture toughness is also derived by use of matrix crack initiation measured in the experiment. The pseudo-values of fracture toughness are calculated based on the energy release rate analysis referred to in the Analysis section. The temperature dependence, together with the absolute toughness values, are in good agreement with the results from DCB specimens. This strengthens the reliability of fracture

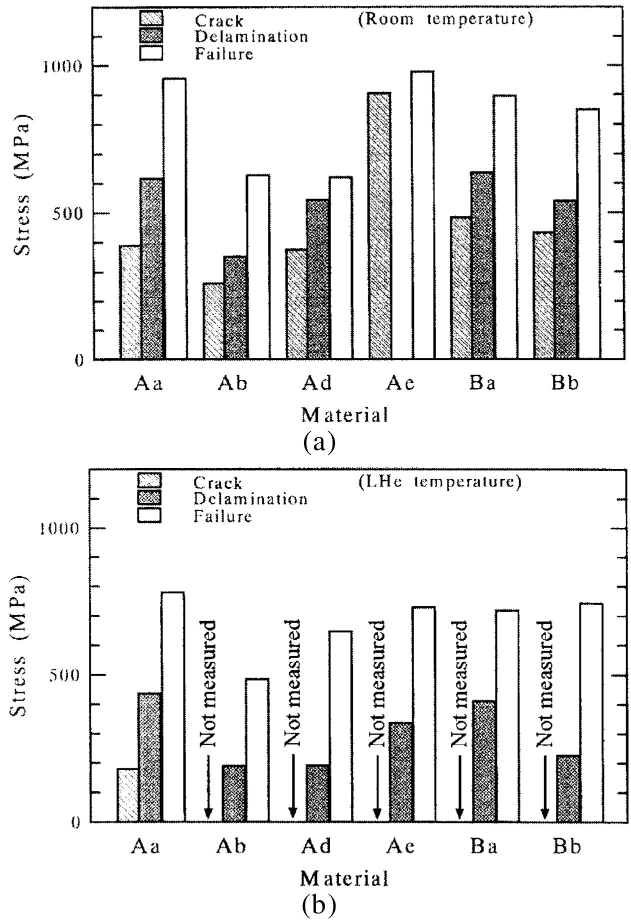


**Figure 6.** Stresses at damage initiations and final failure for different material systems. (a) Room temperature. (b) LHe temperature.

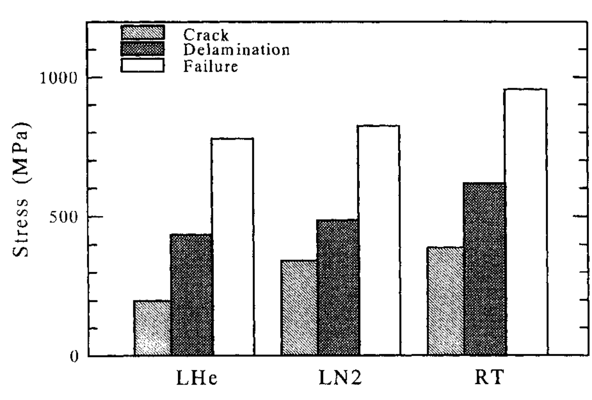


**Figure 7.** Temperature dependency of stresses at damage initiations for type *Aa* material system (IM600/133).

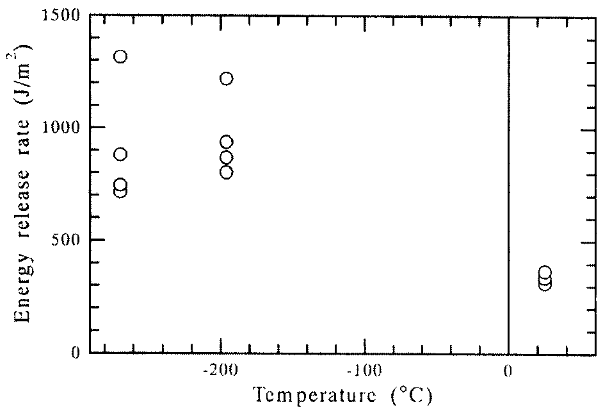


**Figure 8.** Interlaminar fracture toughness measured with DCB specimens (Type *Aa*, IM600/133).

toughness measured with the DCB specimens, showing the increase of the value under cryogenic conditions.

## 5. CONCLUSIONS

The cryogenic performances of different types of CFRPs are experimentally evaluated to survey the basic applicability of the materials to the cryogenic propellant tanks. Temperature dependent material constants, tensile strength, interlaminar fracture toughness are experimentally obtained, together with detailed observations of matrix cracks and delaminations. The results indicate that matrix cracks may be a major critical issue when the current material system is considered for application to the cryogenic propellant tanks. The numerical predictions of the delaminations and matrix cracks are conducted to theoretically support the experimental consequences. The results suggest that possible application of a reliable liner system must also be considered. At the same time, an analytical scheme for evaluating the propellant leakage through matrix cracks existing in multiple layers in laminates must be developed to reduce the usage of liner materials for weight savings.



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