# EFFECTS OF FREEZE-THAW CYCLES ON FRACTURE TOUGHNESS OF ADHESIVELY BONDED CFRP JOINTS

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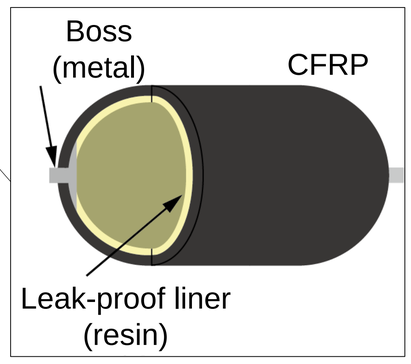
## 1. Introduction

**Carbon fiber reinforced plastics (CFRP) tanks bring many advantages for liquid hydrogen storage.**


Hydrogen-powered aircraft

**Advantages of using CFRP cryogenic tank**
- Lightweight compared to metals
- Passive utilization of anisotropy in design
- Low thermal conductivity to prevent boil-off




CFRP cryogenic tank

Understanding of properties in adhesive parts (e.g., cylinder/boss part) is important considering various factors including environmental effects.

Refueling: Freezing ⟺ Maintenance: Thawing

- Effects of freeze-thaw (FT) cycles must be taken into consideration.
- Repeated FT cycles may trigger damage of adhesive resin due to volumetric expansion of water molecules[2].
- Correlation between fracture mechanisms and mechanical properties of adhesive joints are not fully understood.

**●Objectives**

**Elucidating the effects of FT cycles on fracture toughness of adhesively bonded CFRP joints**

- Mode I fracture toughness tests on moisture-absorbing adhesively bonded CFRP joints after FT cycles
- Tensile tests of bulk adhesive for correlating the property changes of adhesively bonded CFRP joints
- Fracture surface observations of DCB specimens

1) https://www.airbus.com/en/innovation/zero-emission/hydrogen/zeroe, 2) S.K. Mital, et al., NASA/TM, **2006-214346** (2006).

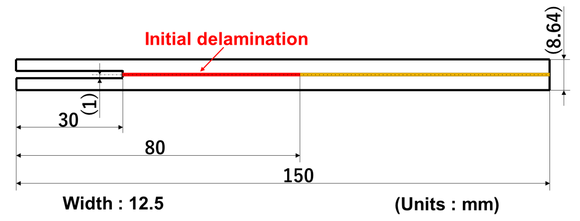
## 2. Experimental procedure

**●Test materials and specimens**

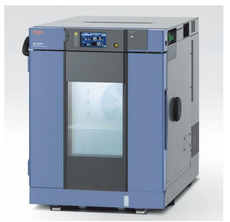
■Specimens for weight change
- Materials: AF163-2M (12.5 × 12.5 × *t*1.2 mm)

■Tensile test specimens of bulk adhesive
- Materials: AF163-2M (90 × 10 × *t*1.2 mm)

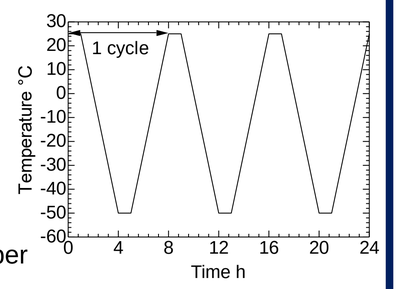
■Double cantilever beam (DCB) specimens
- Adhesive: Structural epoxy adhesive film (AF163-2M, 3M Company)
- Adherends: Unidirectional CFRP (T700S/2592, Toray Industries, Inc.)
- Stacking sequence of the laminates: $[0]_{30}$
- Surface treatment: Sanding
- Initial delamination: 13 μm thick polymer film




**●Procedure for moisture absorption (MA)**
- Maintained for 30 days (720 h) at 60 °C and 95% RH in environmental chamber



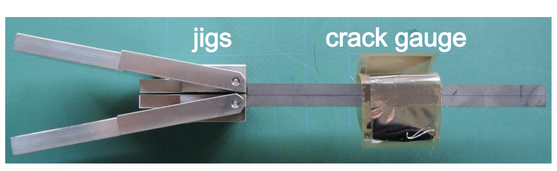
Environmental chamber
SH-662, ESPEC



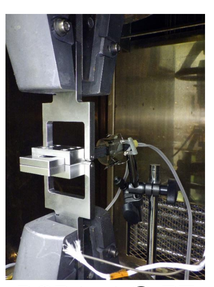

**●Freeze-thaw (FT) cycles**
- Exposed to 75 FT cycles using environmental chamber
- FT cycles consisting of freezing at −50 °C and thawing at 25 °C

| Name of specimens | MA (720 h) | 75 FT cycles |
|---|---|---|
| Control | | |
| MA | ✓ | |
| MA + FT | ✓ | ✓ |

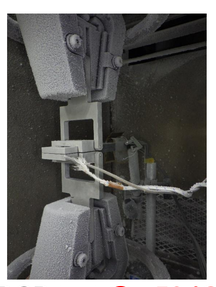
**●Test environment**
- Two temperatures were chosen to consider operating environment (room temperature (RT), −50 °C).




DCB specimen



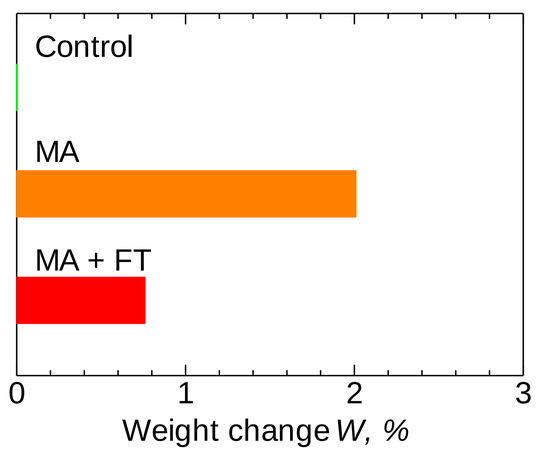
DCB test @ RT



DCB test @ −50 °C

## 3. Results and discussion

**●Weight change of bulk adhesive**

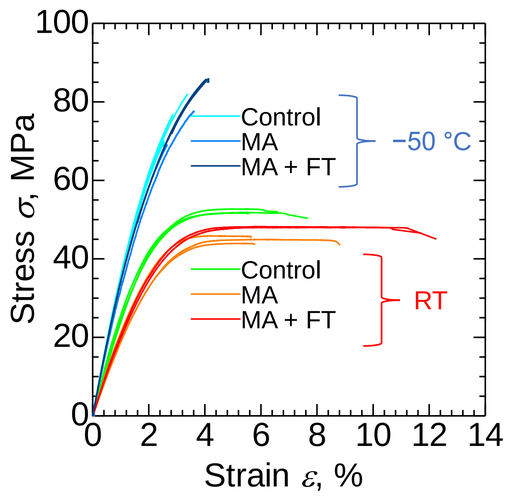



$$W = \frac{m_1 - m_0}{m_0} \times 100$$

$W$ : Weight change (water absorption)
$m_0$ : Mass before water absorption
$m_1$ : Mass after water absorption

➢ **Water possibly evaporated during the FT cycles.**

**●Tensile tests of bulk adhesive**

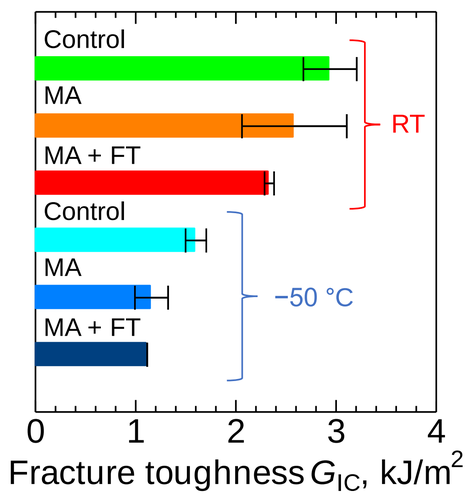



- Ultimate stress decreased due to moisture absorption.
- ➢ **The effect of plasticization by water molecules was observed[3].**
- FT cycles on MA resulted in a slight increase in ultimate stress.
- ➢ **Evaporation of water during FT cycles was possibly caused.**
- Ultimate stress increased but maximum strain at failure decreased significantly from RT to −50 °C.
- ➢ **Changes in bulk adhesive properties at low temperature were observed.**

**Combined effects of MA and FT cycles on mechanical properties of bulk adhesive**

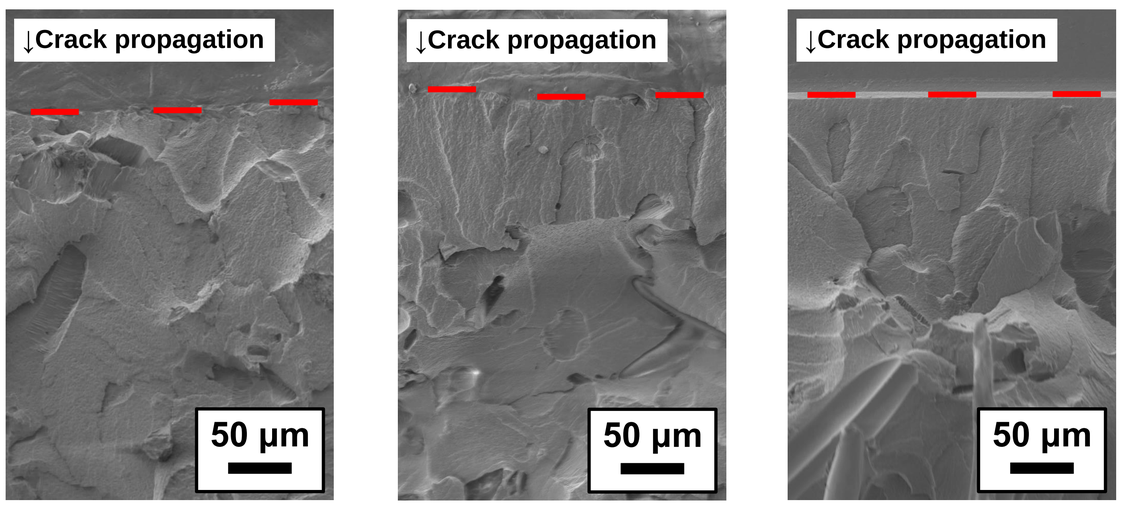
3) K. Kishimoto, et al., ASME, Applied Mechanics Division **187**, pp.11–16 (1994).

**●Mode I fracture toughness**




- Fracture toughness decreased due to moisture absorption.
- ➢ **Fracture toughness of MA specimens reflected bulk adhesive properties (decrease in ultimate stress).**
- Further decrease in fracture toughness occurred due to FT cycles although increase in ultimate stress and maximum strain at failure were observed in bulk adhesive.
- ➢ **Unlike bulk adhesive, the effects may be irreversible in adhesively bonded joints.**
- Fracture toughness decreased at −50 °C.
- ➢ **Decrease in the maximum strain of bulk adhesive at −50 °C was well reflected.**

**●Fracture surface observations of DCB specimens**




**Control (RT)** **MA (RT)** **MA + FT (RT)**

- Change in fracture surface was not observed at micro scale owing to FT cycles.
- ➢ **The effects of FT cycles were due to changes in properties at molecular scale.**

## 4. Conclusions

Mode I fracture toughness tests were conducted to investigate the effect of FT cycles on the mechanical properties of MA adhesively bonded CFRP joints. In addition, the results were correlated with bulk adhesive properties and fracture surface observations. The findings can be summarized as follows.

- **Fracture toughness decreased owing to moisture absorption, and further decreased after FT cycles.**
- **Fracture toughness decreased for all conditions at −50 °C compared to RT results.**
- **Fracture surfaces remained unchanged after MA and FT cycles.**

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