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# A novel experimental technique to determine the tack development during automated placement of uncured thermoset carbon/epoxy tows



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#### ABSTRACT

In-situ bond strength toughness (IBST), commonly referred to as tow tackiness, is a first order property affecting the adherence quality and defect formation (e.g., wrinkles, folds) during automated tow placement (ATP) processing of uncured thermoset polymer matrix composite (PMC) tows. Tow-tow IBST develops over characteristic millisecond timescales ( $t_c \le 50ms$ ) due to the rapid tow placement velocity of  $\sim 1$  m/s. In this paper, an experimental technique is presented to determine millisecond timescale tow-tow mode-I IBST in terms of fracture mechanics-based traction-separation relationship. The test specimen consists of two tows bonded to rigid platens with a pre-crack between them to induce crack initiation. Experiments are performed using IM7-G/8552 carbon/epoxy prepregs for both long and short timescales at a constant debonding rate of 5 mm/s, contact pressure of 0.23 MPa, and bonding temperature of 40 °C. In addition, high-speed two-dimensional digital image correlation (2D-DIC) is used to image and measure the tow-tow interfacial deformation during debonding. The peak traction and apparent energy release rate are found to significantly decrease (about 60 %) at the short millisecond timescale contact hold times compared to longer (i.e., second) timescale.

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## 1. Introduction

Automated tow placement (ATP) is a complex manufacturing technique that utilizes narrow composite prepreg tows to produce composite structures [1,2]. The ATP process requires rapid application of temperature and pressure, and the process conditions can result in defects (wrinkles, folds) that are known to be detrimental to composite bond strength and can lead to premature component failure. Previous studies have shown that tow tackiness or in-situ bond strength-toughness (IBST) is a first-order parameter affecting adherence quality for a bonded layer [3–5]. The typical contact time available for bond formation,  $t_c$ , in ATP manufacturing operations, is quite small ( $t_c \le 50~ms$  millisecond-scale) and wrinkle defect formation times are much smaller.

Even though IBST is developed on a short time scale, a common approach to characterize bond 'tow tackiness' is to perform tensile probe tack testing [6,7]. This method relies on maximum vertical debonding force as the metric at timescales much larger than  $t_c$ 

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and does not include a pre-crack to induce crack initiation for the determination of critical energy release rate,  $G_{IC_i}$  from the traction separation curve [7,8]. In the enclosed paper, a novel experimental technique is introduced to determine short millisecond timescale Mode-I IBST for uncured thermoset tows.

## 2. Experimental methodology

#### 2.1. Materials

In this study, a commercially available IM7-G/8552 uncured carbon epoxy prepreg (areal weight 190  $\rm g/m^2$  and thickness 0.16 mm) is used.

### 2.2. Experimental setup

Fig. 1 shows the experimental setup to determine IBST under controlled conditions for temperature, pressure, and contact time. In this setup, the bottom platen is secured to the fixed base of the Instron 5944 single-column machine, while the top platen is attached to a 2 kN load cell to apply compaction pressure for bonding and tensile force for separation. Heating of tows is achieved

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using two 250 W/120 V MR16 halogen bulbs of 2-inch diameter. Fig. 2(a) shows a schematic of the IBST test specimen that includes two carbon fiber epoxy IM7-G/8552 tows that were stored at -18 °C and thawed for 1.5 hours till they reached room temperature. The tows are 40 mm long and 6.35 mm wide each, along with a pre-crack to induce crack initiation. The two tows are attached to the two steel platens using Loctite 426 adhesive. A 0.0127 mm thick optically clear FEP film made of Teflon fluoroplastic is used to create a 25 mm long pre-crack and a bonding contact area of  $15 \times 6.35 \text{ mm}^2$ . Millisecond timescale contact hold time ( $t_c$ ) for bonding is achieved using a combination of displacement-control and force-control modes in the Instron test system (see Fig. 2(b) and (c)). The procedure begins with the top platen being brought close to the bottom platen until there is a 10 mm gap between them. A stopwatch is prepared to record the time (8 min) it takes to heat the tows to 40 °C. The heating lamps and stopwatch are started simultaneously and stopped as soon as 40 °C is reached marking the beginning of the test. There is a 2-second delay between turning off the lamps and initiating the test. Three repeat tests are performed for  $t_c$  = 30 ms, 50 ms, and 1 s, respectively.

#### 3. Results and discussion

#### 3.1. Short millisecond contact hold time bonding

As shown in Fig. 2(c),  $t_c$  refers to the actual force hold time over which the applied pressure  $(P_c)$  corresponding to the contact force  $(F_c)$  is constant. All the representative tests are conducted at bonding temperature,  $T_b = 40$  °C and  $P_c = 0.23$  MPa. Test is performed at  $t_c = 1$  s for  $b_r = 0.1$  mm/s and  $db_r = 5$  mm/s and for  $t_c = 30$  ms and 50 ms tests are conducted at a bonding rate  $(b_r)$  of 0.5 mm/s and a debonding rate  $(db_r)$  of 5 mm/s with all other variables held constant. It is observed that, when increasing  $b_r$  from 0.1 mm/s to 0.5 mm/s,  $F_c$  of 30 N (input to Instron 5944) doubled to 60 N due to inertial effects. For  $t_c = 1$  s, experiments are performed for  $b_r = 0.1$  mm/s and  $db_r = 5$  mm/s. The force versus time during compression and hold stages are shown in Fig. 3(a) for both the short and long  $t_c$ .

Fig. 3(b) shows the traction-separation response during debonding at different timescales. The fracture mechanics-based traction separation relationship is determined in terms of the crack tip stress and crack initiation displacement, with the area under the curve representing the critical energy release rate. Since loading is applied along the entire length of the specimen, it is assumed

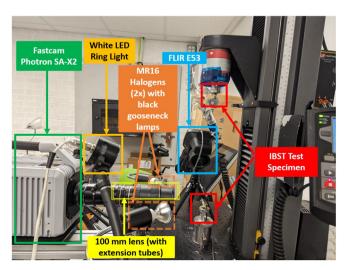
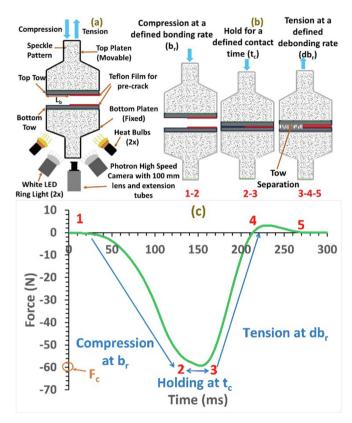


Fig. 1. Experimental setup for tow-tow IBST measurement.

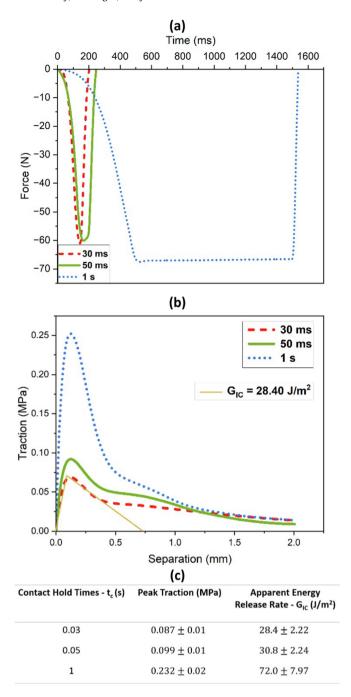


**Fig. 2.** (a) Schematic for tow-tow IBST test specimen with bond length  $L_b$ , high-Speed 2D DIC, and heating setup, (b) IBST Test Procedure, (c) A schematic force versus time for IBST test at a  $t_c$  = 30 ms,  $b_r$  = 0.5 mm/s,  $db_r$  = 5 mm/s,  $P_c$  = 0.23 MPa, and  $T_b$  = 40 °C.

that tractions are uniform along the interface and are calculated as force over interfacial area. Since the tow specimens are so thin (thickness of 0.16 mm), it is difficult to visualize the instant of crack initiation and directly measure the crack tip displacements as the tows separated. Therefore, displacements are measured on the platens using high-speed 2D-DIC and it is found that the displacement measured by 2D-DIC is identical to the crosshead displacement. As expected, the peak traction significantly decreases with decreasing contact hold time. The average peak traction at a short timescale of 30 ms (0.087 MPa) is 62.5 % lower than the peak traction at a longer timescale of 1 s (0.232 MPa). Based on previous studies [8], an apparent critical energy release rate  $(G_{IC})$  is calculated by assuming a linear softening beyond the peak traction, as shown in Fig. 3(b) for  $t_c$  = 30 ms. Fig. 3(c) lists the average peak traction and  $G_{IC}$  for all the tests. Similar to peak traction,  $G_{IC}$  also significantly decreases with decreasing contact hold time. The average apparent  $G_{IC}$  at a short timescale of 30 ms (28.4 J/m<sup>2</sup>) is 60.6 % lower than the apparent  $G_{IC}$  at a longer timescale of 1 s  $(72.0 \text{ J/m}^2)$ .

### 3.2. Mechanisms of bond formation and separation failure modes

From Fig. 3(c), interface bond formation mechanisms between two uncured thermosetting polymer tows due to the application of rapid pressure and temperature (temperature below cure initiation, no chemical crosslinking) is a strong function of contact hold-time,  $t_c$ . Since it is known that uncured thermosets initially consist of long chain molecules with weak bonds, the application of pressure results in improved contact through the deformation of asperities that enhances factors such as interdiffusion [3,5]. It should be noted that temperature dependent polymer relaxation time,  $t_p$  is



**Fig. 3.** (a) Force versus time during bonding, (b) Traction separation response at  $P_c$  = 0.23 MPa, and  $T_b$  = 40 °C, (c) summary of the average IBST values for the representative tests.

on the order of seconds. For  $t_c \ll t_r(T_b)$ , experimental data in Fig. 3 (c) suggests that IBST in the short millisecond timescales is contact-controlled. Though bonding when  $t_c > t_r$  will be primarily due to interdiffusion that is nominally independent of pressure, when bonds are formed at timescales where  $t_c \ll t_r$ , they are strongly influenced by contact-controlled pressure. Fig. 4 shows the post-test fracture surface images captured for tests conducted at  $t_c = 30$  ms and 1 s. For  $t_c = 30$  ms, Fig. 4 (left), shows minimal fiber pullout with a predominantly adhesive failure at the towtow interface. As shown in Fig. 4 (right), significant fiber bridging in the form of cohesive failure is observed at the tow-tow interface for  $t_c = 1$  s since increasing  $t_c$  results in polymer interdiffusion that enhances bond formation.

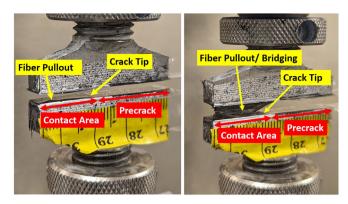


Fig. 4. Post-test fracture surface image (left) short timescale (30 ms), (right) long timescale (1 s).

#### 4. Conclusions

A novel experimental technique is introduced to measure short timescale mode-I IBST or tackiness of uncured thermoset tows. The test specimen consists of two tows bonded to rigid platens with a pre-crack to induce crack initiation. Mode-I IBST tests are performed at short millisecond contact hold times of 30 ms and 50 ms and at long contact hold time of 1 s while maintaining  $T_b = 40~{\rm ^{\circ}C}$  and  $P_c = 0.23~{\rm MPa}$ , with IBST quantified by the peak traction and measured  $G_{IC}$ . The peak traction and the apparent  $G_{IC}$  are found to decrease by 62.5 % and 60.6 %, respectively, as  $t_c$  is reduced from 1 s to 30 ms. Adhesive failure is observed at the tow-tow interface for  $t_c = 30~{\rm ms}$  and cohesive failure is observed within the tow for  $t_c = 1~{\rm s}$ .

## **CRediT authorship contribution statement**

**Debrup Chakraborty:** Formal analysis, Investigation, Methodology, Validation, Visualization, Writing – original draft. **Karan Kodagali:** Formal analysis, Investigation, Methodology. **Sreehari Rajan-Kattil:** Methodology, Writing – Review & Editing. **Dennis Miller:** Investigation, Methodology. **Subramani Sockalingam:** Conceptualization, Supervision, Writing – Review & Editing, Project administration, Funding acquisition. **Michael A. Sutton:** Writing – Review & Editing, Funding acquisition, Resources.

## **Declaration of competing interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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