Effect of an Atmospheric Pressure Plasma Treatment on the Mode I Fracture Toughness of a Co-Cured Composite Joint

1.Introduction

•Convenient to co-cure the two materials using single processing process

Pros:

- Reduces cost
- Reduces processing time

Cons:

- Epoxy resin contains moisture(free and bounded) → interfacial failure

- To eliminate the drawback:
    - Apply plasma treatment on the surface → Too much treatment is detrimental

- Most paper focuses plasma treatment on second bonding composite

- This paper investigates the effect on co-cured joints

2.Manufacture&Methods

Materials:

- 177 °C cure unidirectional carbon-fiber/epoxy prepreg (CYCOM 977-2/HTS)
- Dual 120/177 °C cure epoxy film adhesive (FM300-2M)

Each substrate consists 10plies of 977-2/HTS prepreg

Specimen:

Width:25mm Length:150mm Thickness:5.6mm Initial Crack Size:45mm

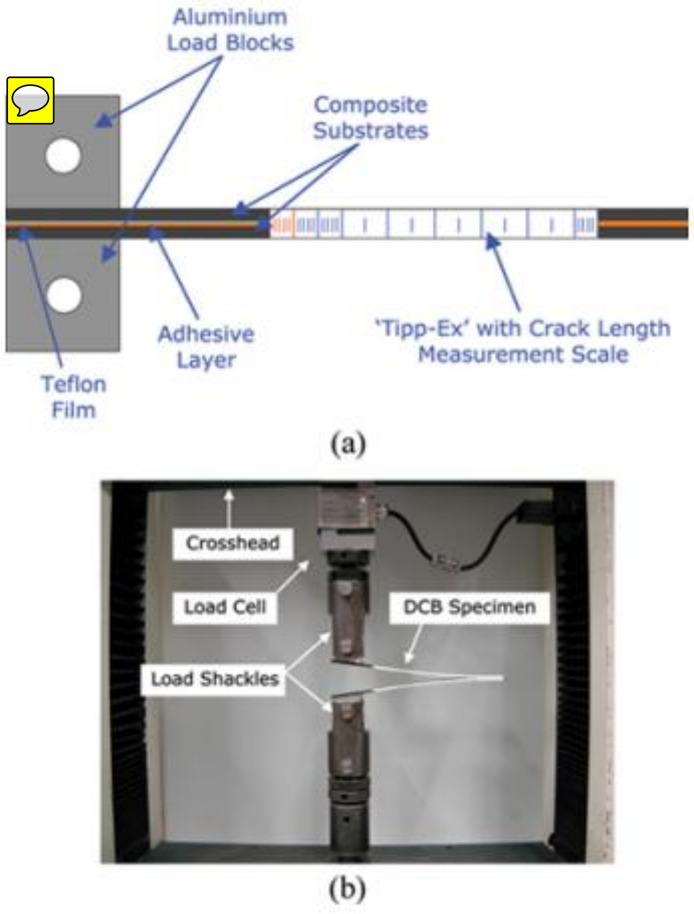
2.1 Fracture Test Methods

- Double cantilever beam test with British Standards 7991
- Constant crosshead displacement: 1mm/min
- Three repeats on each specimen

2.2 Scanning Electron Microscopy Method

2.3 Thermal Characterisation Methods

- Heated from 25°C to 350°C (10 °C /min)
- To determine glass transition temperature



Fracture Test Method

2.4 Surface Energy Characterisation

- Sessile drop technique
- Surface energy is calculated with OWRK method.

2.5 Labline Plasma Treatment System

- Constant speed: 1.5 m/min
- Applied only on two prepreg surface that direct contact with film adhesive

2.6 Optical Emission Spectroscopy

- To analyze plasma content
- Always N2 peaks in spectrum(diffusion of air into plasma)
- He/O2 leads more interaction with prepreg → higher surface energy

2.6 X-Ray Photoelectron Spectroscopy

- To study chemical composition of treated surfaces
- Mean of 3 measurements on different spots

3 Result & Discussion

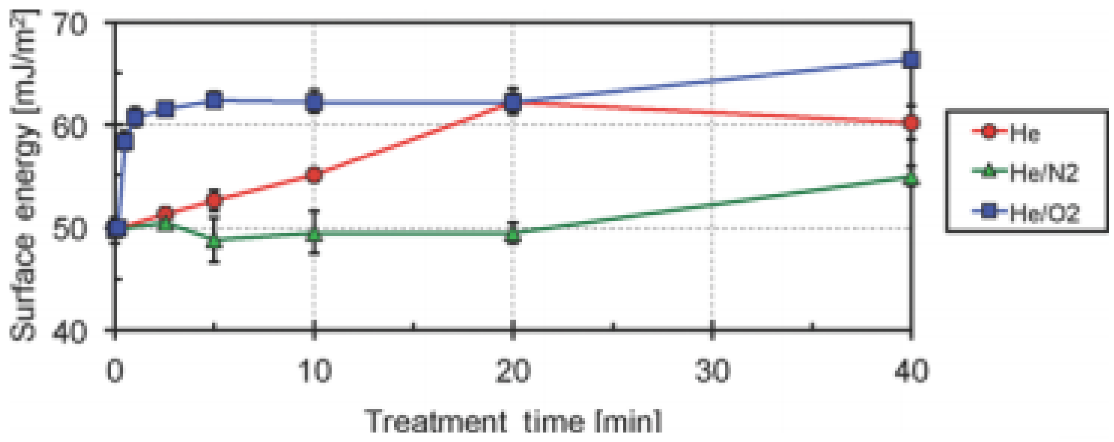
<u>3.1 Surface Energy Analysis</u>

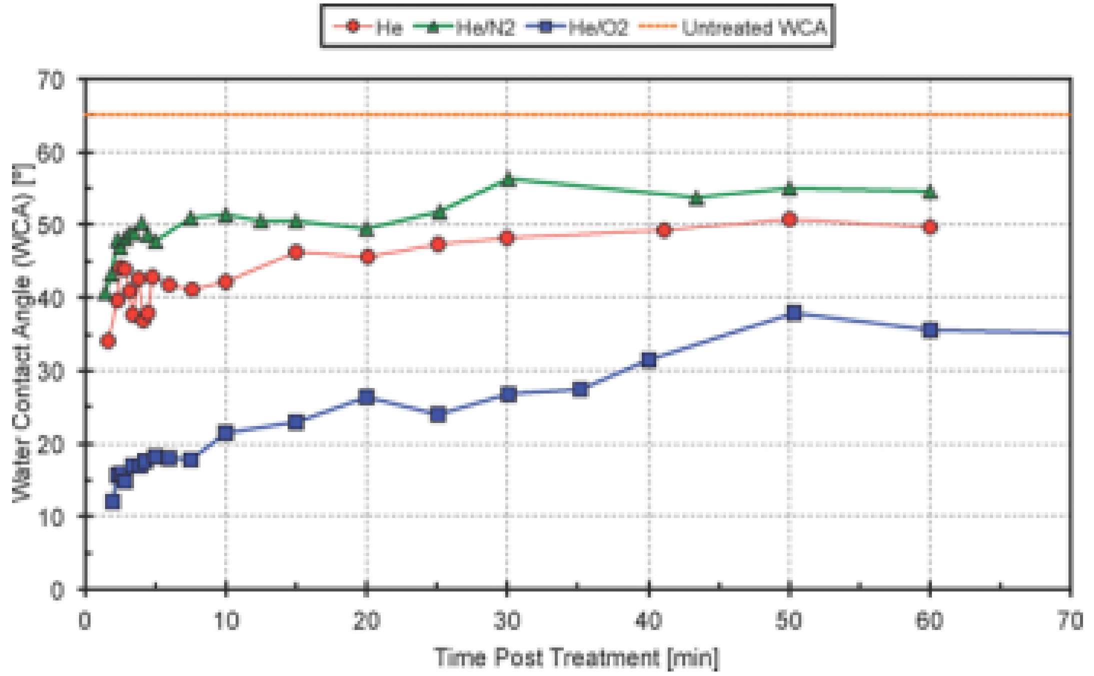
Three gas mixtures:

- He
- He/N2
- He/O2

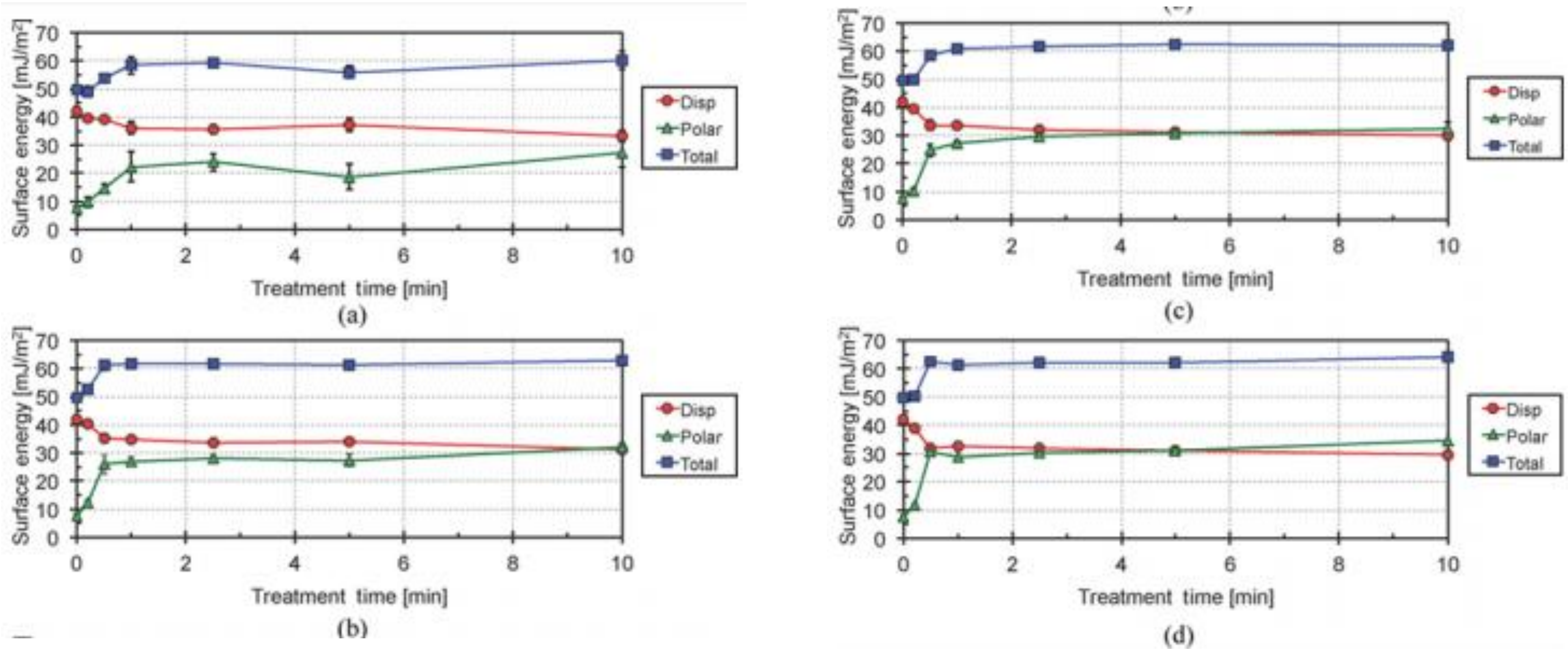
Power is fixed at 1200W

Maintained at 10L/min

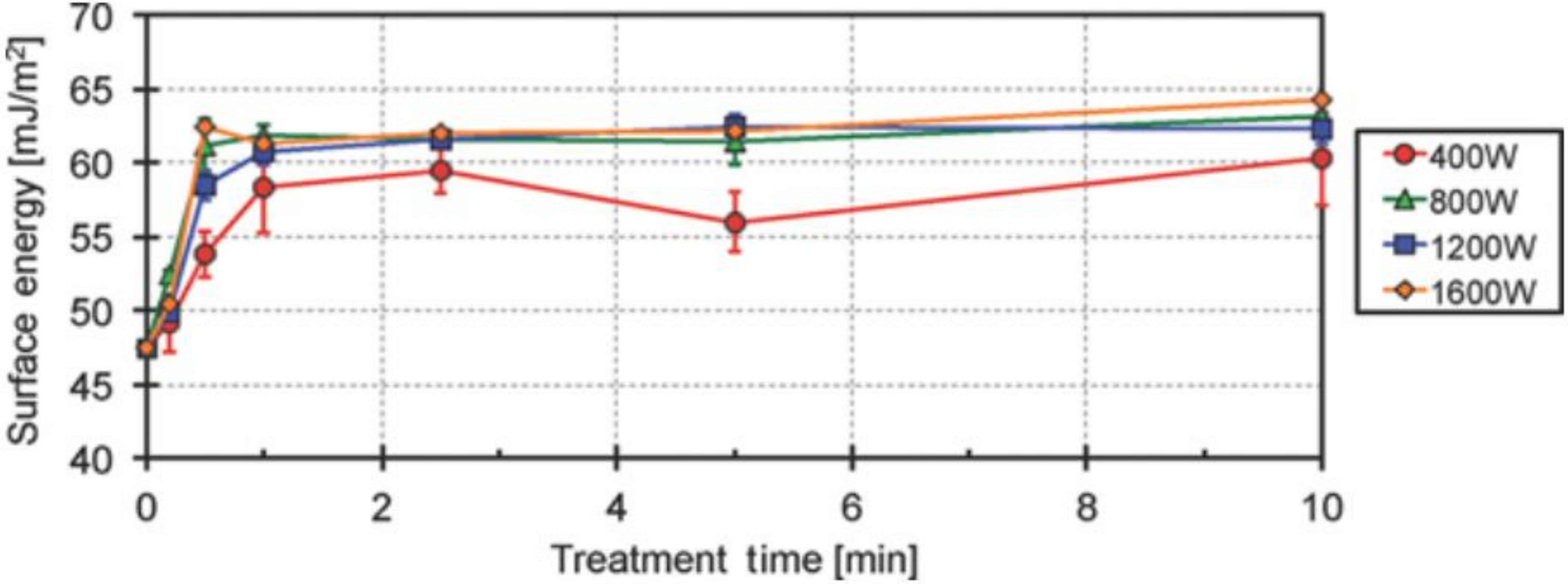




•Different power levels are also applied.



He/O2 plasma treatment at
(a) 400 W, **(b)** 800 W, **(c)** 1200 W, and **(d)** 1600 W



Surface energy [mJ/m²]
70
65
60
55
50
45
40
0
2
4
6
8
10
Treatment time [min]
400W
800W
1200W
1600W

3.2 Mode I Fracture Toughness

- He/O2 treatment at 1600W
- Exhibits largest increase in surface energy at 2.5 min

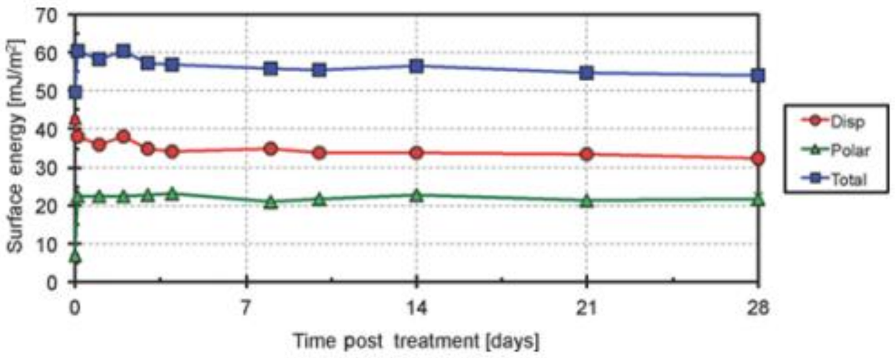


Figure 10 Surface energy of prepreg treated with a He/O_2 plasma up to 28 days after treatment. Note that in some cases, the error bars are obscured by the data points.

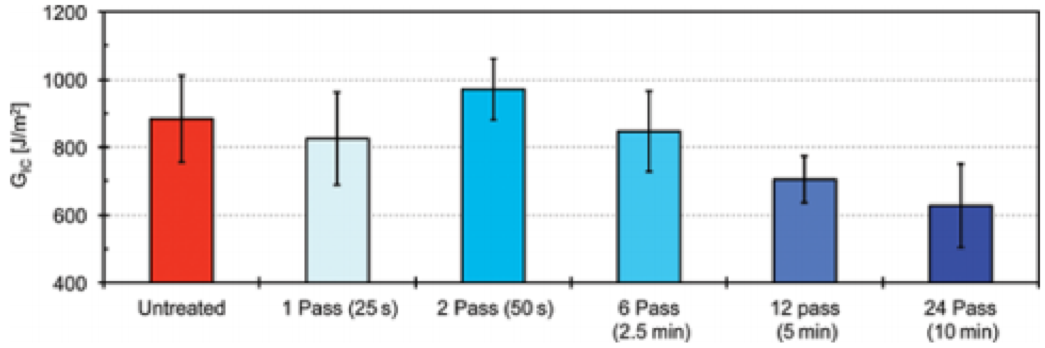


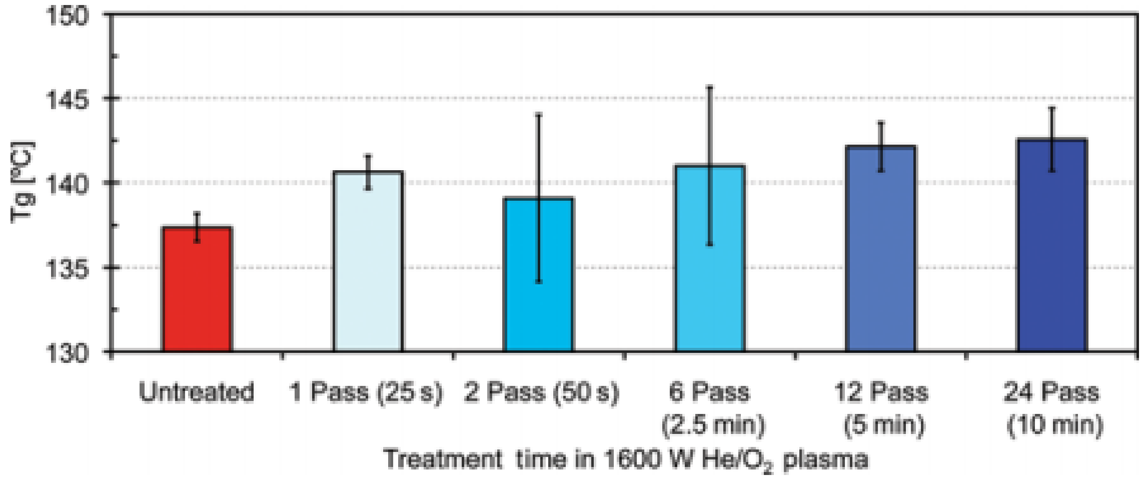
Figure 11 Mode I fracture toughness, G_{IC}, of co-cured joints treated with a He/O_2 plasma. The applied power was constant at 1600 W for all treatments.

3.3 XPS Analysis

- Samples that treated with He/O2 at 1250W for 5 phase
- The presence of sulphur (thermoplastic toughening agent or agent in resin)
- Silicon concentration is in decrease (contamination)

3.4 Microscopy and Thermal Analysis

- Long treatment → reduced fracture toughness in co-cured joints
- Higher concentration of voids weakens the interface and reduces toughness
- Glass temperature of the adhesive increases
- Excess plasma treatment ablates and oxidizes →Morphological changes in material



4 Conclusion

- He/O_2 treatment is effective in increasing surface energy of prepreg.
- He and He/N_2 is very inefficient in increasing the surface energy.
- Excessive treatments leads severe degradation in the performance under Mode I.
- Highly polar surface attracts moisture to the interface.
- Long plasma treatment causes pitting → reduction in fracture toughness

Mixed-Mode Fracture Toughness of Co-Cured and Secondary Bonded Composite Joints

1 Introduction

- Drilling & bolding processes weakens the composite material

- Superior method of using adhesive may improve stress distribution.

- Co-cured joints suffer from trapped water inbeing released during the process.
    - Affects the quality of the adhesive joint

- An increase in the Mode I component increases Mode II percentage.

- Aims is to link the experimentally measured fracture toughness with observed mechanism in the joint.

2 Materials & Manufacture of Joints

- 180 °C cure unidirectional carbon-fibre/epoxy prepreg (CYCOM 977-2/HTS)
- A dual 120/180 °C cure epoxy film adhesive (FM300-2M)
  - Contains polyester scrim cloth with random orientation

- Produced in-house, using vacuum bagging layup procedure
  - 120 °C or 180 °C
  - Each consists 10 plies → grit-blastered to remove release agent and roughen
    - CC180 = Co-cured at 180 °C
    - SB180 = Secondary bonded at 180°C
    - SB120 = Secondary bonded120°C

3 Experimental Methods

3.1 Fracture Test Method:

- Nominal Width = 25mm
- Length = 150mm (DCB) and 190mm (ADCB/ELS)
- Initial Crack Starter = 45mm(DCB) and 6mm(ADCB/ELS)
- Total Thickness ~ 5.6mm

- **Mode I Fracture Toughness:**

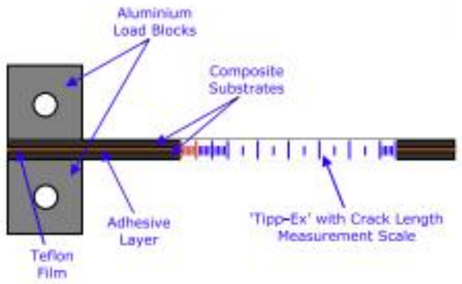
using double cantilever beam test (DCB)

- **Mode II Fracture Toughness:**

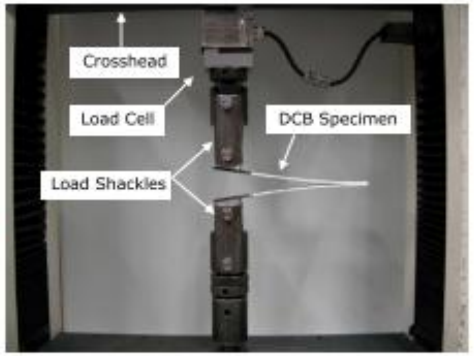
using calibrated-end-loaded-split (C-ELS)

- **Mixed Mode:**

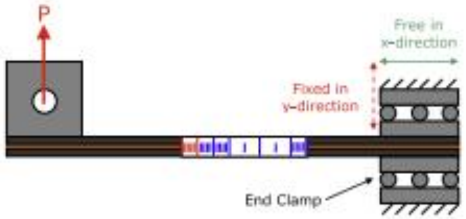
using asymmetric double cantilever beam (ADCB)



(a) DCB illustration.



(b) DCB photo.



(c) ADCB illustration.

(d) ADCB photo.

(e) ELS illustration.

(f) ELS photo.

- LEFM Method is applied → To create sharp crack.
    1. Loaded until crack propagates to 5mm
    2. Unload
    3. Then resume the loading

- Crack length is monitored with x10 magnification.
- Constant crosshead displacement rate of 1mm/min

- Mode II crack propagation is unstable → ratio of crack length (a/L) > 0.55

3.2 Microscopy Method

- Optical Microscope and Scanning Electron Microscope after fracture test

3.3 Thermal Characterisation Methods

- Sample is heated from 25 °C to 350 °C (at rate of 10 °C /min)
- To determine the glass transition temperature of the adhesive

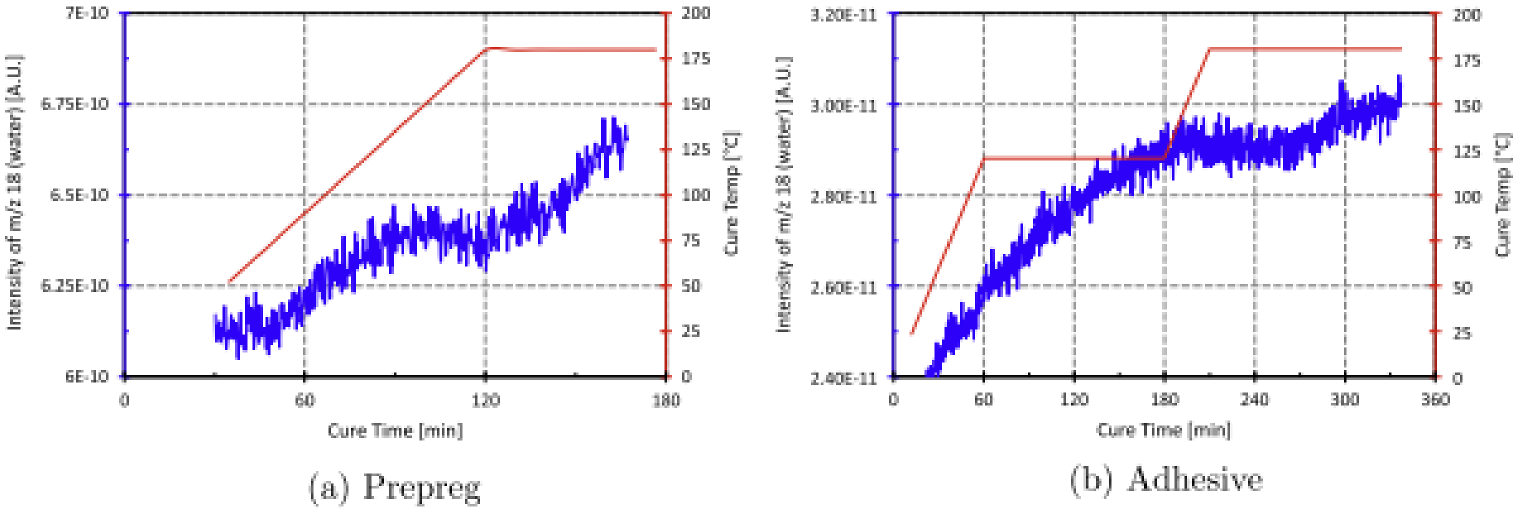


Fig. 2. TGA-MS of water released (blue trace) from prepreg & adhesive during the course of a heating regime (red trace). The ramp rate in (a) is 1.5 C/min while both ramps in (b) are 2 C/min. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

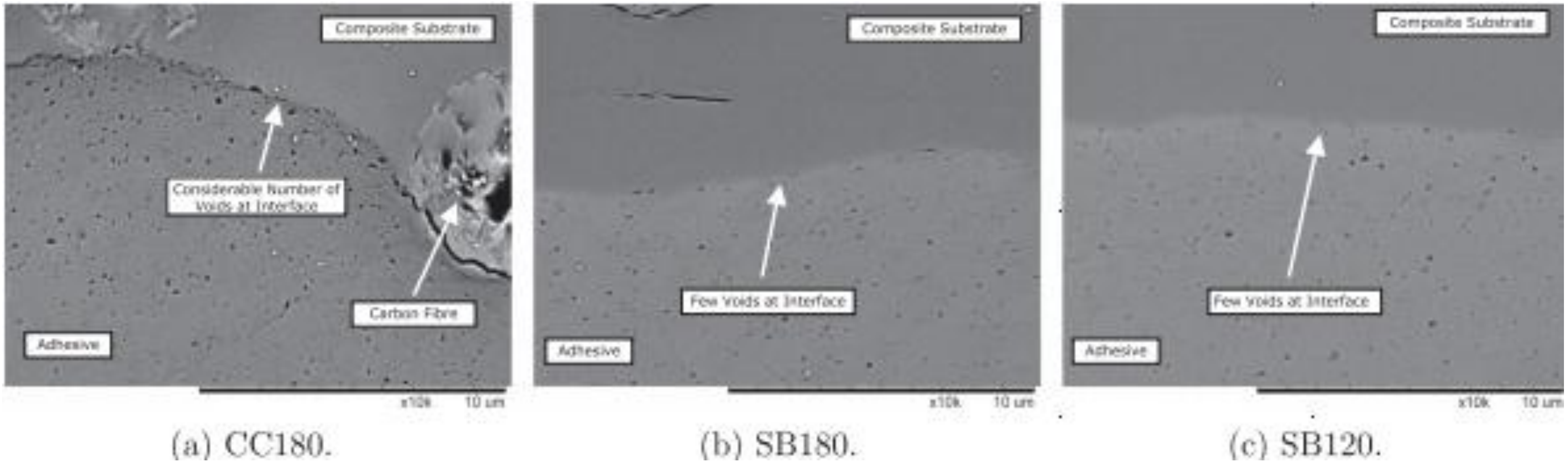


Fig. 3. Adhesive–substrate interface of each joint system. Magnification is 10,000.

4 Result &Discussion

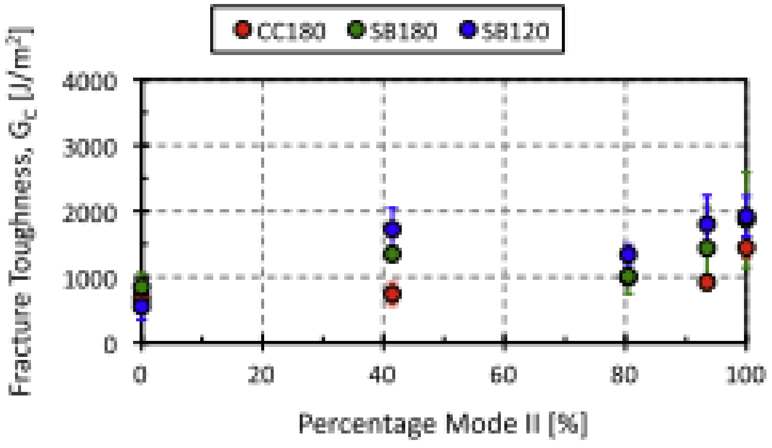
4.1 Initiation Fracture Toughness

- Teflon film not giving a consistent starter defect

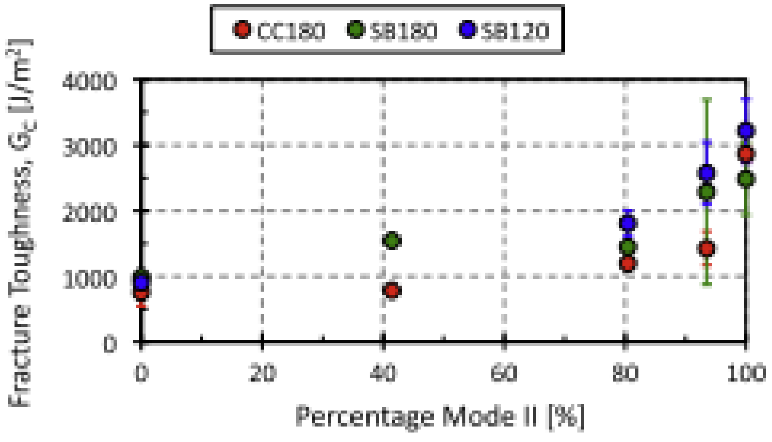
4.2 Propagation Fracture Toughness

- R-curve behavior is occurred.
- Fracture energy increases with higher percentage Mode II.
- Pure Mode II fracture energy reaches 5-6 times of Mode I.

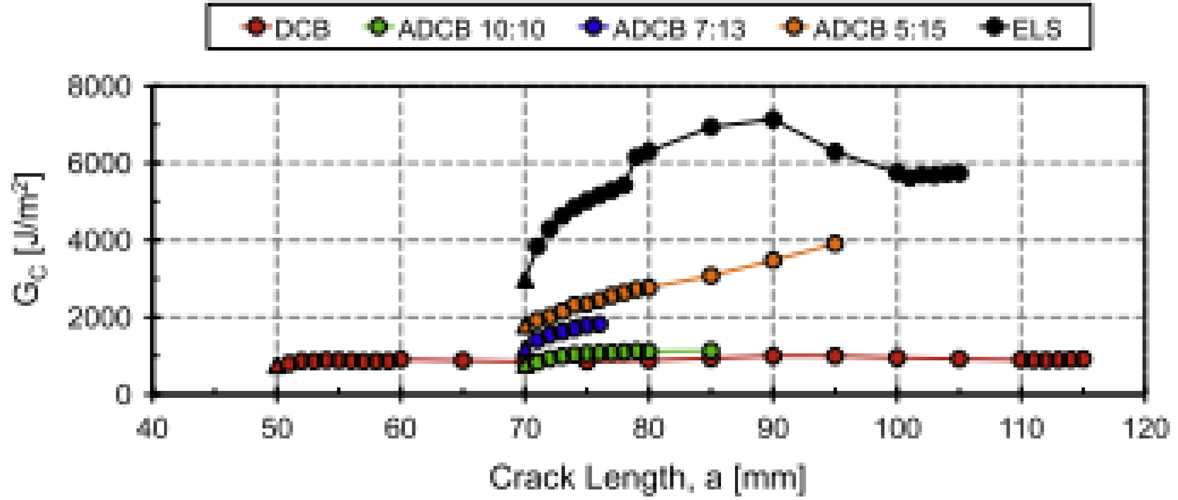
- CC180 joint exhibits interfacial failure.
- Secondary bonded failed either cohesively or interlaminarly
- Interfacial failure in several CC180 in Mixed-Mode → by voids at the interface
- Interlaminar failure in some SB180 and SB120 → grit-blastered surface



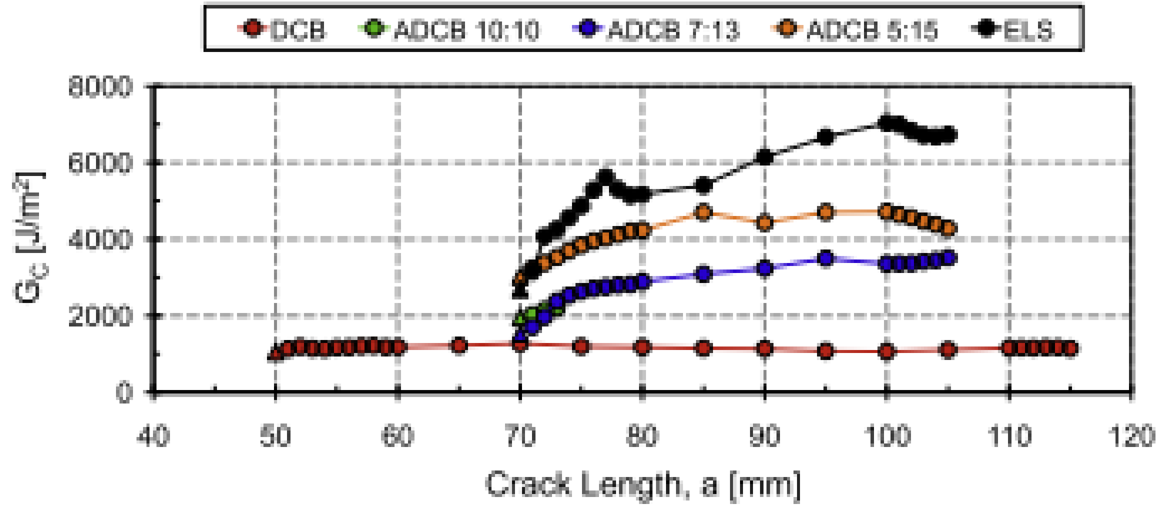
(a) Testing from PTFE insert.



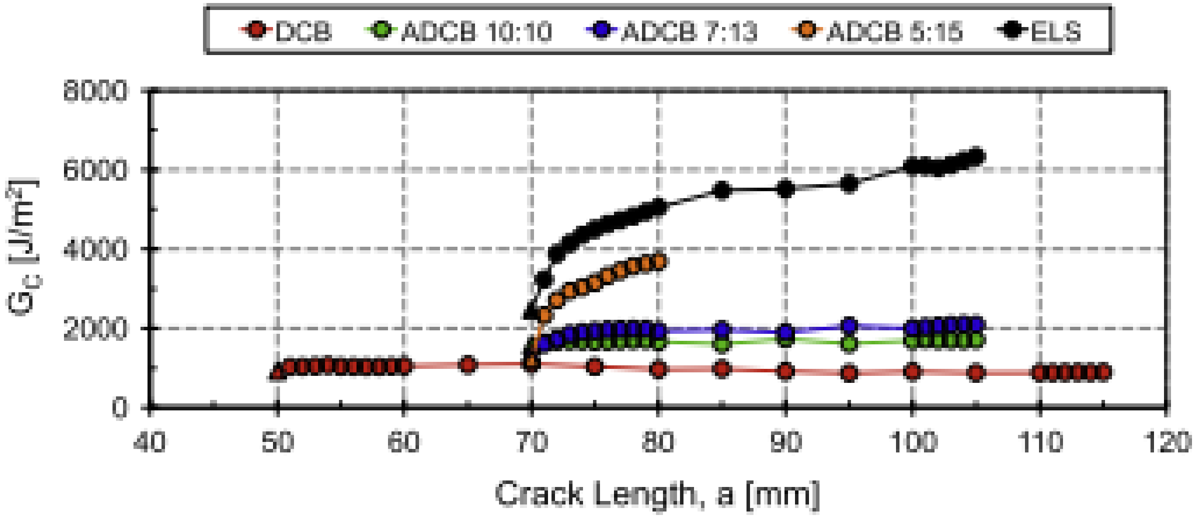
(b) Testing from precrack.



(a) CC180.



(c) SB120.



(b) SB180.

4.3 Failure Criteria

- Experimental data fits Benzeggagh failure criteria

Table 1
Constants used in Benzeggagh (α in Eq. (4)) and Hashemi (κ and φ in Eq. (5)) failure criteria.

Joint system	CC180	SB180	SB120
G_{IC} (J/m^2)	883	1009	1124
G_{IIC} (J/m^2)	5011	5728	5817
α	16	7.90	4.7
κ	7.45	3.67	2.01
φ	−5.40	−5.92	−3.62

- CC180 is by far the weakest
- SB120 is the toughness

4.4 Thermal Analysis

- Curing conditions affect the glass transition temperature of the adhesive

Expected T_g:

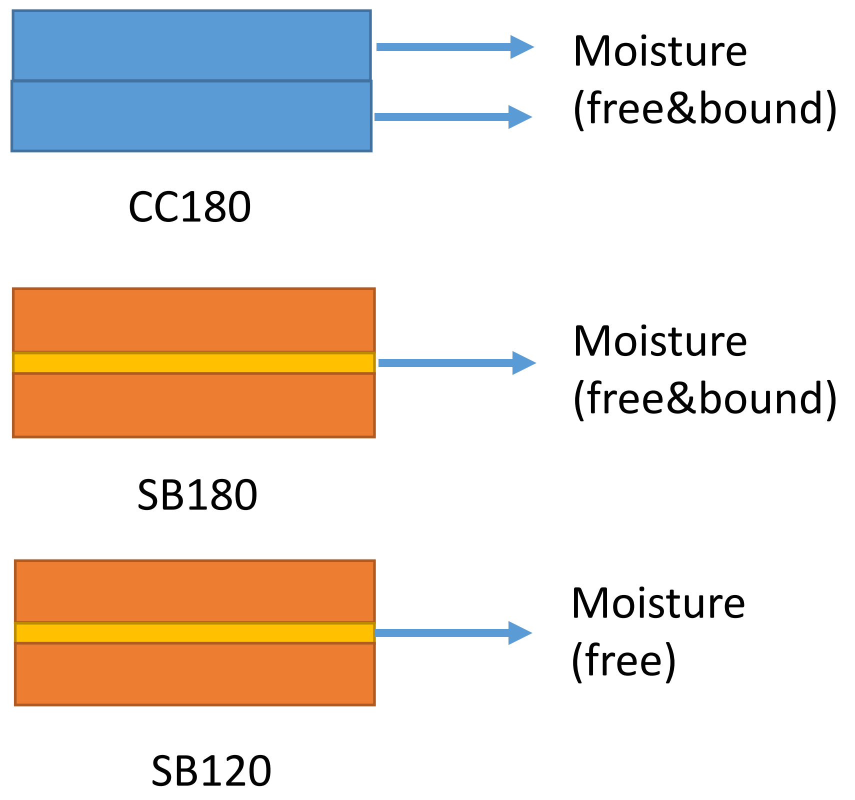
- SB180>SB120>CC180

Resultant T_g:

- SB120>SB180>CC180

Resultant G_{Ic} & G_{IIc}:

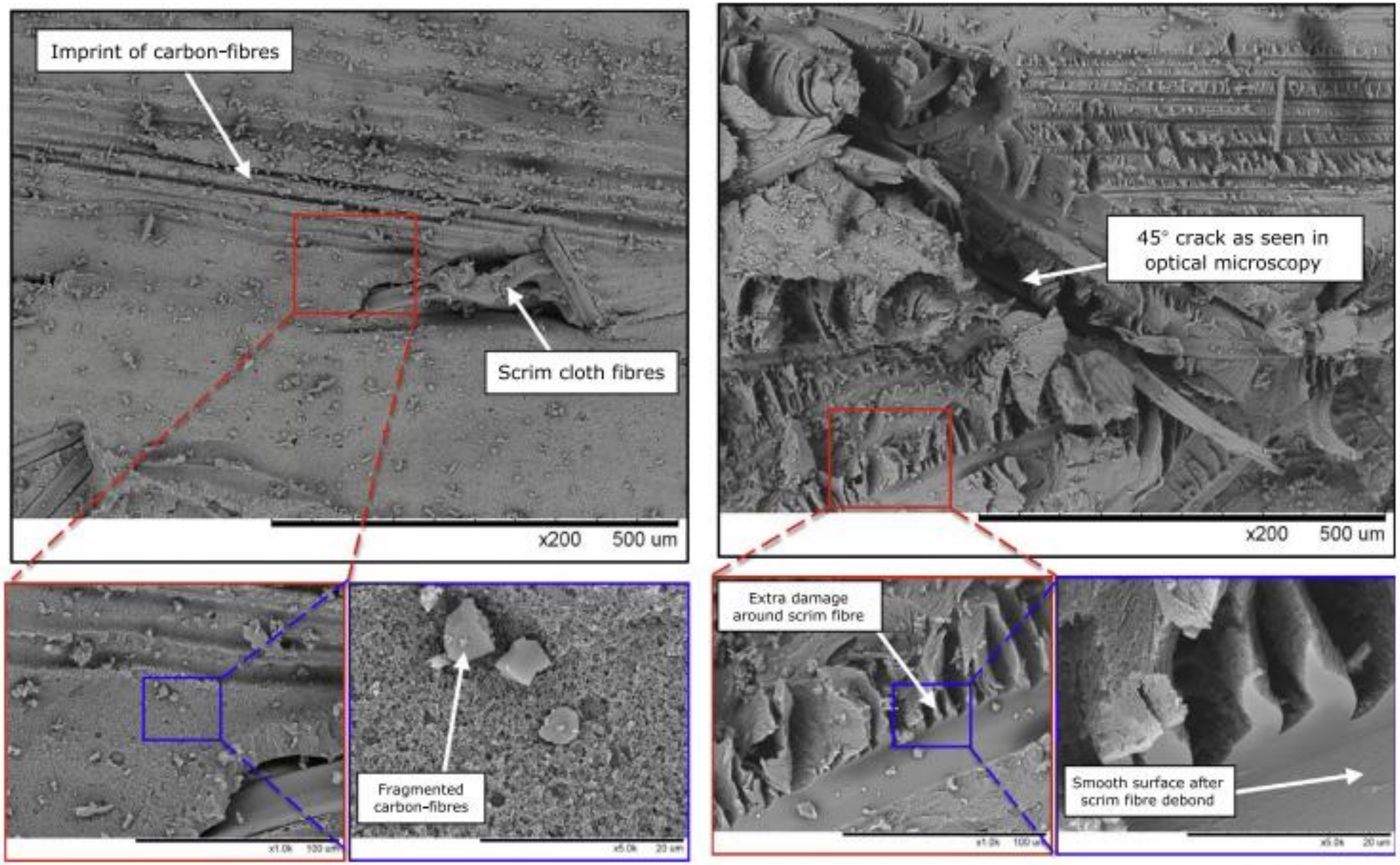
- SB120>SB180>CC180



- <u>4.5 Increase in Surface Area:</u>
- The direction of crack propagation is from left to right
- Mode II increases → Crack path exhibits jagged profile

- <u>4.6 R–Curve Behavior:</u>
- Particularly under pure Mode II loading
- Crack length of ~15mm is needed for steady state G_{IIC}

- <u>4.7 SEM Analysis of Fracture Surface:</u>
- Large 45° cracks and debonded scrim cloth fibres are observed
- As fibre debonds, stress free region is created in adhesive layer



(a) Mode I

(b) Mode II

Fig. 21. SEM of mode I & II interfacial fracture surface of CC180 joint system. In (a) & (b), the top image is $\times200$ magnification. Bottom left and right are $\times1000$ and $\times5000$ respectively. Note that (a) was taken in a region were the adhesive remained entirely on one side of the joint.

5 Conclusion

- Fabrication methods causes variation in material toughness.

Co-cured process: plasticize polymer and introduce voids

Secondary bonding: water already is released

  - SB180 affected by free and bonded water
  - SB120 affected by free water

- Mode II increases → Total Fracture Energy, G_C, also increases
- **CC180:** cracks are forced to weakened interface (due to voids) under Mixed-Mode
- **SB120:** Toughest due to cohesive failure and contribution of fibre pull-ou

- Fracture surface area increases about 1.6-1.8 →Not factor in increase of Mode II toughness
  - Believed due to increasing damage zone, associated with adhesive plasticity

- R-curve behavior is due to expanding damage zone
  - Zone is found at least 15mm long

- CC180 is quite weak under Mixed-Mode
  - But under Mode II, its toughness can be comparable with others
  - But small percentage of Mode I is needed to crack