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Combining Through-Thickness Reinforcement and Self-Healing for Improved Damage Tolerance and Durability of Composites

T. Kevin O'Brien, Michael W. Czabaj, and Jeffrey A. Hinkley
Langley Research Center, Hampton, Virginia

Spiros Tsampas and Emile S. Greenhalgh
Imperial College, London, United Kingdom

Gregory McCombe, Ian P. Bond, and Richard Trask
University of Bristol, Bristol, United Kingdom

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Introduction

A well-established scheme for mitigating or suppressing delamination growth in a composite laminate is to insert carbon Z-pins in the through-thickness direction [1,2]. The apparent interlaminar fracture toughness of the composite is increased due to the tractions imposed by the Z-pins as they pull out during delamination growth, thereby providing improved damage tolerance [3,4]. Other attempts have been made to introduce self-healing resins to the crack plane as delaminations grow, to restore some of the initial fracture toughness. This may be accomplished via microencapsulation [5-7] or embedding the resin in hollow glass tubes in the planes of the fibers [8-10]. Introducing a self-healing resin has the added benefit of sealing the crack plane to prevent moisture ingression, and hence, improving durability.

A study was undertaken to develop a prototype method for adding through-thickness hollow glass tubes infused with uncured resin and hardener to a carbon Z-pin through-thickness reinforcement field. The Z-pins are intended to provide delamination resistance as they pull out during a delamination event. Simultaneously, the glass tubes will fracture and release the enclosed resin and catalyst. The tractions provided by the Z-pin pull-out mechanism should allow time for the resin to cure, and minimize crack opening to reduce the volume that needs to be filled, resulting in a self-healed fracture plane.

An “initial study” was first undertaken where half the carbon Z-pins were replaced by the hollow glass tubes in the foam preform prior to insertion in the uncured laminate. Because the survival rate of the glass tubes was not sufficient, a more detailed “main study” was then conducted where all of the carbon Z-pins in the preform were inserted into the uncured laminate, and then half were removed and replaced with hollow glass tubes. This technique resulted in a nearly 100% survival rate for the hollow glass tubes, allowing resin insertion, followed by testing, to evaluate the proposed concept.

Materials and Specimen Preparation

Z-pin pre-forms consisting of 0.5 mm diameter pultruded carbon fiber tows embedded in a low density foam were purchased from Albany Engineered Composites. The Z-pins are typically inserted in an uncured tape laminate using an ultrasonic hammer (fig.1), resulting in a 2% by weight areal density of through-thickness Z-pins. For this study, half of the Z-pins in the pre-form were replaced with 0.55 mm outer diameter, 0.4 mm inner diameter, hollow glass tubes.

In the initial study, the tubes were placed in the foam (fig.2) replacing some Z-pins before insertion into three uncured 32-ply, 305 mm square, unidirectional IM7/8552 carbon epoxy prepreg panels. The panels contained a 13 micron thin Teflon film inserted along the middle of the panel at the laminate mid-plane, to create Double Cantilever Beam (DCB) interlaminar fracture toughness specimens. The panels were cured in a press, using the manufacturer’s recommended curing cycle, and then cut into 152 mm long by 20 mm wide coupons using a water cooled diamond saw. The panels were C-scanned to insure no manufacturing flaws were present. Each panel yielded five DCB specimens, for a total of 15 specimens. Loading blocks were then bonded to the specimens using a room temperature adhesive.

In the main study, a second panel was manufactured using 24 plies of unidirectional IM7/8552 carbon fiber epoxy prepreg tape material. As before, a 13 micron thin Teflon film was inserted along the middle of the panel at the laminate mid-plane to create DCB interlaminar fracture toughness specimens. The Z-pin field was inserted first in the uncured laminate, then every other row of Z-pins was removed. Hollow glass tubes were then manually inserted immediately following insertion of a sewing needle of similar diameter in the hole vacated by the Z-pins before relaxation of the resin that would tend to close these holes. Aluminum plates were used to support the unreinforced portion of the plate and prevent tube breakage during cure in the press (fig.3). The panel was cured in the press per the manufacturer's specifications. Inspection of the panel after cure indicated nearly 100% survival of the glass tubes (fig.4). However, the thickness in the Z-pin field was larger than the remainder of the panel due to the lack of local compaction during cure.

The self-healing resin chosen was Product #307 from Epoxy Systems, Inc. of Dunnellon, Florida. This is a very low viscosity, two component, liquid epoxy resin system designed to be mixed at a 2-to-1 ratio of epoxy to hardener (fig.5). At room temperature, the resin-hardener mixture was observed to gel in less than 30 minutes. The manufacturer indicates that the mixture cures tack-free in five hours and is fully cured in seven days. The resin and hardener were colored with 1% by weight of commercially available soluble fluorescent dyes for enhanced visibility within the glass tubes and on the composite crack faces. Solvent orange 63 and solvent yellow 98 dyes were selected for the resin and hardener, respectively, because they mix well with epoxy components and fluoresce brightly under long-wave ultraviolet light [11]. When mixed, they yield a maroon color to the epoxy and an orange color to the hardener (fig.5).

Before cutting specimens from the 24-ply panel, the two-part epoxy was injected into the tubes in the region where the first six coupons were located, with part A (resin) filling two neighboring tubes and part B (hardener) filling the adjacent single tube. Prior to cutting these first six DCB specimens, the ends of the tubes were sealed using a liquid rubber cement to insure that the resin and hardener did not escape (fig.6). Coupons that were 152 mm long by 20 mm wide were cut from the panel using a diamond saw at a slow rate to prevent heat build up and to eliminate the need for a coolant and prevent moisture contamination (fig.7). The specimen width was chosen so that at least six tubes were present across the specimen width. Piano hinges were then bonded to the specimens using a Hysol 9460 epoxy. The adhesive was cured at room temperature for two days. X-ray tomography was performed on one specimen (3B) to illustrate the variability in pin and tube placement and the disturbance in the fiber straightness resulting from pin and tube insertion. Figure 8 shows the resin and hardener insertion pattern superposed on the X-ray image of the 24-ply DCB specimen. For each DCB specimen cut from the panel, each row of glass tubes had at least two sets of three tubes with the desired 2-to-1 ratio of resin and hardener. The remaining specimens were cut from the 24-ply panel before inserting the resin and hardener.

Testing and Post-mortem Examination

Initial Study

Specimens were cut from the three 32-ply panels and were tested without healing agents. A total of 15 specimens were tested. DCB tests (fig.9) were conducted according to ASTM D5528 [12]. Figure 10 shows a typical load–displacement plot with four distinct load drops corresponding to pull out of the four rows of carbon Z-pins. Optical and scanning electron microscopy were used for fractographic examination of the fracture plane in the vicinity of the hollow glass tubes. Fractography indicated that the tubes did fracture as the delamination grew in the DCB specimens while the Z-pins pulled out (fig.11a). However, it was evident that some of the tubes were either crushed during the insertion process (fig.11b), or broke and then filled with prepreg resin that bled during the cure cycle (fig.11c). Examination of all 15 specimens indicated that only 21-29% of the hollow glass tubes had survived the Z-pin insertion process and the panel curing cycle. Hence, no attempt was made to fill these specimens with healing agent.

Main Study

DCB tests were conducted according to ASTM D5528 [12]. Tests were run in a 5-kip hydraulic load frame in displacement control at a rate of 0.04 in/min (1 mm/min). Load was monitored via a 100 lbf (0.44 kN) capacity load cell mounted in series with the 5-kip (22.4 kN) load cell. During the test, delamination growth was observed on the specimen edges using a pair of traveling video cameras. Digital video images were saved periodically using a laptop computer that also captured the load and displacement associated with each saved video frame. The first three specimens tested (3A, 2A, and 3B) were from the initial group of six specimens that were filled with resin and hardener, in the 2-to-1 ratio prescribed, and sealed with rubber adhesive before being cut from the panel as described earlier.

The first DCB coupon cut from the 24-ply panel (specimen 3A) was loaded until delamination grew past all four rows of tubes (fig.12). Two digital cameras recorded the progression of delamination on the two specimen edges during the test (fig.13) and the load displacement trace was recorded and plotted (fig.14). The specimen was unloaded, then clamped shut for 24 hours. The specimen was reloaded to determine if the adhesive had released from the tubes and healed the crack plane. The first reloading load-displacement curve followed the original unloading curve up to the load where unloading began in the initial test, indicating no healing had occurred (fig.14). Hence, either the resin did not flow out of the tubes once the delamination had passed, or the volume of the resin and hardener in the sealed tubes was not sufficient for adequate mixing and healing of the delamination fracture plane. The specimen was then reloaded a second time to grow the delamination to separate the crack planes for inspection.

The second DCB coupon cut from the 24-ply panel (specimen 2A) was loaded until delamination grew past two rows of tubes. The specimen was unloaded, then clamped shut for 24 hours. The specimen was reloaded to determine if healing had occurred. The reloading load-displacement curve followed the original unloading curve up to the load where unloading began in the initial test, indicating no healing had occurred (fig.15). Hence, either the resin did not flow out of the tubes once the delamination had passed, or the volume of the resin and hardener in the sealed tubes was not sufficient for adequate mixing and healing of the delamination fracture plane. A wedge was inserted between the DCB arms before unloading. The sealing resin and hardener were mixed in the prescribed 2-to-1 ratio and injected directly into the crack plane with a hypodermic needle (fig.16). The DCB arms were clamped shut for 4 days. The specimen was then reloaded a second time to determine if the cured resin and hardener recovered the original toughness. The second reloading load-displacement plot followed the slope of the original test matching the original stiffness, and delamination began at approximately 80% of the original load, indicating that healing did occur (fig.15). Hence, the premixed healing resin bonded well with the 8552 base resin along the fracture plane once both the resin and hardener were mixed in the correct proportions and manually placed in the needed area.

The third DCB coupon cut from the 24-ply panel (specimen 3B) was loaded until delamination grew past all four rows of tubes, unloaded, then clamped shut for 24 hours. The unloading curve was not plotted because a wedge was inserted between the DCB arms before unloading to allow the healing resin and hardener opportunity to mix and fill the crack plane. The specimen was reloaded to determine if the healing resin and hardener had indeed released from the tubes and healed the crack plane. The first reloading load-displacement curve had a significantly lower slope than the initial loading curve, following what may have been the original unloading curve, up to the load where unloading began in the initial test, indicating no healing had occurred (fig.17). Next, the sealing resin and hardener were mixed in the prescribed 2-to-1 ratio and injected directly into the crack plane with a hypodermic needle. The DCB arms were clamped shut for 24 hours. The specimen was then reloaded a second time to determine if the cured resin and hardener recovered the original toughness. The second reloading load-displacement plot followed the slope of the original test matching the original stiffness, and delamination began at approximately 125% of the original load, indicating that healing did occur (fig.17). Hence, the premixed healing resin chosen bonded well with 8552 base resin along the fracture plane once both the resin and hardener were mixed in the correct proportions and manually placed in the needed area. The increased crack initiation load compared to the original test may simply reflect the thicker bond line of the sealed delamination due to the inability to completely close the original crack grown through four rows of tubes and pins.

The remaining specimens tested were cut from the panel before any resin was injected into the tubes. The fourth DCB coupon cut from the 24-ply panel (specimen 11B) was initially tested without adhesive in the glass tubes. The specimen was loaded until delamination grew past the first two rows of tubes, then unloaded (fig.18). A wedge was inserted between the DCB arms, and the dye-enhanced healing resin and hardener were premixed in the prescribed 2-to-1 ratio and injected into the glass tubes with a hypodermic needle (fig.19). Ultraviolet light image of the specimen surface indicated that the healing resin was injected into a majority of the tubes, and in addition, covered the 8552 prepreg resin rich areas around the tubes and z-pins (fig.20). The DCB arms were clamped shut for 48 hours. The specimen was then reloaded to determine if the cured resin and hardener recovered the original toughness (fig.21). The reloading load-displacement plot followed the slope of the original test, matching the original stiffness. Delamination began at approximately 110% of the original load, indicating that healing did occur (fig.18). Hence, the self-healing resin chosen was clearly bonding to the fracture plane once both the resin and hardener were mixed in the correct proportions and manually place in the needed area via the glass tubes. The increased crack initiation load compared to the original test may simply reflect the thicker bond line of the sealed delamination due to the inability to completely close the original crack grown through two rows of tubes and pins. Loading was continued in an attempt to grow the delamination past the last two rows of tubes. Observations of the specimen edges during the test showed evidence of secondary branch delamination planes forming (fig.22). Nearly twice as much load, compared to the initial loading, was required to extend the delamination past the last two rows of carbon Z-pins. At this point the specimen failed completely. Post-mortem examination of the fracture surface indicated the healing resin had also adhered to the previously pulled out Z-pins (fig.23). This was most likely a result of the pre-mixed resin filling the cracked resin rich areas around the tubes and z-pins (fig.20). Hence, repeated loading had to overcome not only the sealed fracture plane, but the additional tractions necessary to pull out the Z-pins a second time as well.

The fifth DCB coupon cut from the 24-ply panel (specimen 7B) was initially tested without adhesive in the glass tubes. The specimen was loaded until delamination grew past the first two rows of tubes, and was then unloaded (fig.24). A wedge was inserted between the DCB arms and the dye-enhanced healing resin and hardener were injected in-situ into the glass tubes separately using a hypodermic needle in an alternating pattern in each row, similar to that shown in figure 8. The DCB arms were clamped shut for 48 hours. The specimen was then reloaded to determine if the cured resin and hardener recovered the original toughness. The end of the specimen was clamped just beyond the fourth row of Z-pins to prevent complete separation of the DCB specimen, and the delamination was grown through all four rows of glass tubes. The reloading load-displacement plot followed the slope of the original unloading curve up to the end of the first two rows of tubes, indicating that no healing had occurred in the first two rows (fig.24). Hence, there was insufficient resin flow, mixing, and/or crack opening for healing to occur at this point. The specimen was held at the maximum load and the additional resin and hardener were injected in-situ using a hypodermic needle to mimic the additional resin supply available had a secondary reservoir of resin and hardener been attached to the tubes. Individual droplets of resin and hardener were observed inside the opened delamination plane at the specimen centerline (fig. 25). The specimen was unloaded and monitored via the traveling video cameras. Movement of the resin and hardener in the tubes was observed, filling the delamination plane as unloading occurred. Some resin was clearly visible on the edge once the specimen was completely unloaded and clamped (fig.26). The DCB arms were clamped shut for 48 hours. The specimen was then reloaded for a second time to determine if the in-situ injected healing resin and hardener cured and recovered the original toughness. The second reloading load-displacement plot followed the slope of the loading portion of the first reloading curve, indicating that healing may have occurred, at least in the vicinity of last two rows of tubes (fig. 27). Hence, in-situ injection of resin and hardener directly into glass tubes performed at maximum load after initial fracture resulted in partial healing of the fracture plane. However, the maximum load never reached the level of the initial test, or first reloading test, before the specimen fractured into two pieces. Furthermore, during the test there was some evidence of uncured resin leaking from the tubes (fig.28). Ultraviolet images of the fracture surface indicated the presence of resin over much, but not all, of the delamination plane (fig.29).

For the sixth DCB coupon cut from the 24-ply panel (specimen 10B), the dye enhanced sealing resin and hardener were inserted separately in the glass tubes using a hypodermic needle in an alternating pattern in each row as done on the previous specimen. The specimen was loaded until delamination grew past all four rows of tubes, and was then unloaded (fig.30). The DCB arms were clamped shut for 48 hours. The specimen was then reloaded to determine if the cured resin and hardener recovered the original toughness. The reloading load-displacement plot followed the slope of the original unloading curve, indicating that no healing had occurred (fig.30). Hence, there was insufficient resin flow, mixing, and/or crack opening for healing to occur at this point. The specimen was held at the maximum load and the additional resin and hardener were inserted in-situ using a hypodermic needle to mimic the additional resin supply available had a secondary reservoir of resin and hardener been attached to the tubes. Some resin was visible on the edge once the specimen was completely unloaded and clamped (fig.31). The DCB arms were clamped shut for 48 hours. The specimen was then reloaded for a second time to determine if the in-situ injected resin and hardener reacted and recovered the original toughness. During the test there was some evidence of uncured resin leaking from the tubes (fig.32). The second reloading load-displacement plot had a slope in between the initial loading and the first reloading curve, indicating that some healing may have occurred (fig.33). Hence, in-situ injection of resin and hardener directly into glass tubes performed at maximum load after initial fracture resulted in partial healing of the fracture plane. In addition, the maximum load exceeded the level of the initial loading test, and nearly recovered the maximum load observed during the first reloading test. The specimen experienced significantly higher displacements before it eventually fractured into two pieces. Ultraviolet images of the fracture surface indicated the presence of resin over much, but not all, of the delamination plane (fig.34).

Results and Discussion

For the initial 32-ply panels, good bonding was achieved between the 8552 base resin and the glass tubes. During DCB tests, the carbon pins pulled out and glass pins fractured as the delamination progressed. However, microscopic examination indicated that the majority of glass tubes had cured resin inside from the manufacturing process. Examination of the fracture plane indicated glass tube survival rates of only 21-29% for the 15 specimens produced from the three panels via direct insertion of pins and tubes in the preform.

For all specimens tested from the 24-ply panel, manufactured via direct insertion of pins followed by pin removal and manual insertion of glass tubes, good bonding was also achieved between the 8552 base resin and the glass tubes with nearly a 100% survival rate for the glass tubes. However, this tube insertion process was labor intensive and would require automation for practical application. During the DCB tests, the carbon pins pulled out and the glass pins fractured as the delamination progressed. No healing was observed for tests where the unmixed resin and hardener were initially placed in sealed tubes with limited material available to heal the crack plane. Healing did occur for tests where the resin and hardener were premixed in the 2 to 1 ratio prescribed and injected directly into the crack plane or injected in-situ through the glass tubes and allowed to drain into the crack plane. Hence, the self-healing resin system chosen bonded well with the 8552 base resin in the composite along the fracture plane. In one case, premixed resin may have filled the cracked resin-rich regions near tube insertion locations allowing healing agent to attach to pulled out Z-pins, indicating that additional healing may be possible via coating of pulled out Z-pins. In-situ injection of resin and hardener directly into glass tubes, in a staggered pattern to allow for 2 to 1 ratio mixing, resulted in partial healing of the fracture plane, but only if the injection was performed while the specimen was held at maximum load after initial fracture.

This preliminary study indicated that there is some potential for healing delamination via resin and hardener delivered through a through-thickness network of glass tubes, but only if the tubes are connected to a reservoir where additional material may be injected as needed. Furthermore, maximum effectiveness may only be achieved if the crack plane can be held open long enough for the healing material to flow and mix in the proportions needed prior to closing. A method for pumping resin from an attached reservoir may also be needed, in addition to capillary action, for efficient resin flow and mixing. Furthermore, additional recovery of strength may be possible if a technique was available for applying self-healing material directly to pulled out Z-pins.

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Figures

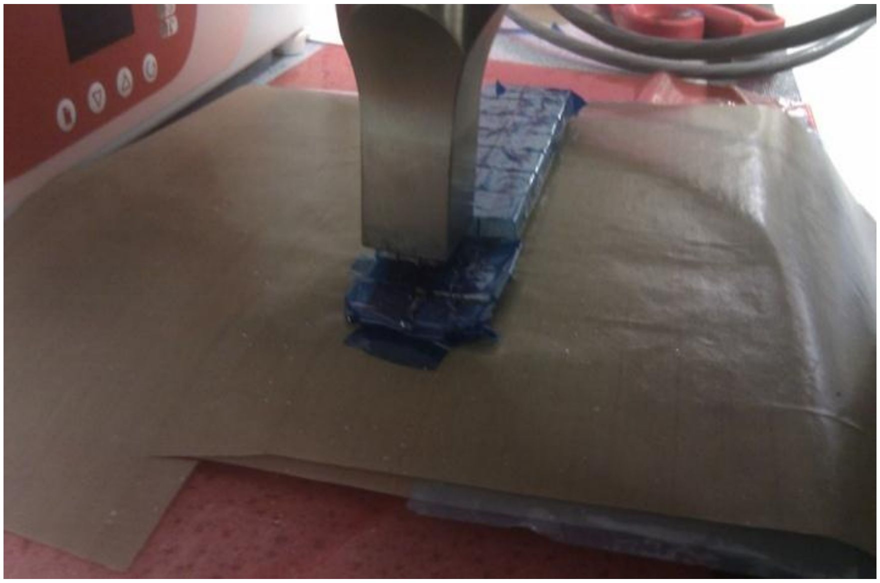


Fig. 1. Z-pin insertion via ultrasonic hammer

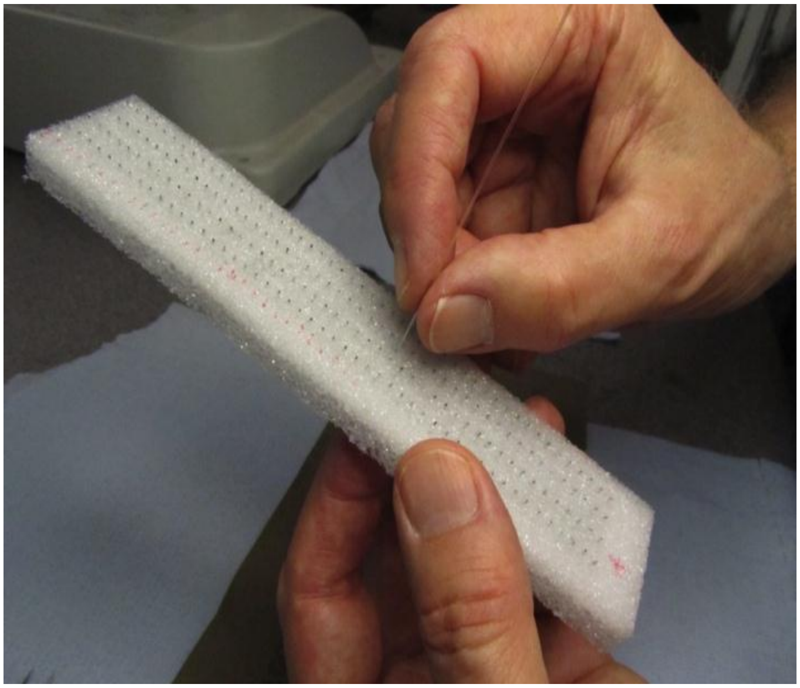


Fig. 2. Z-pin preform with hollow glass tubes

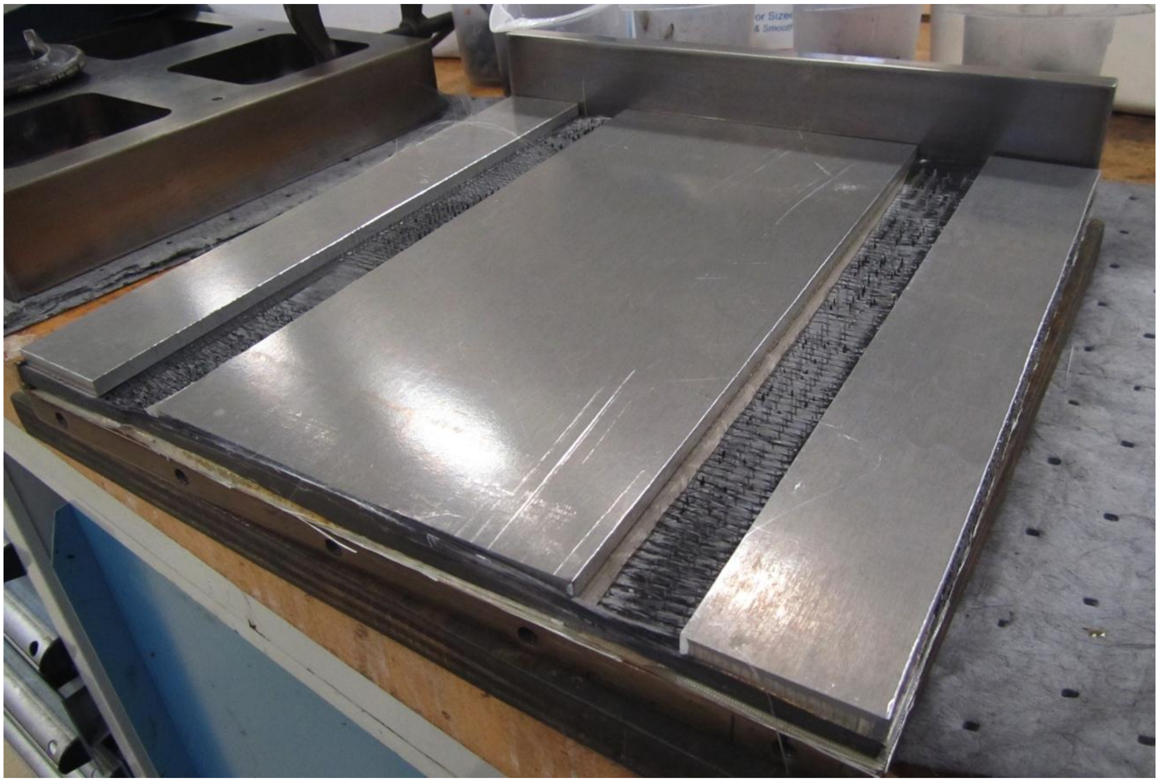


Fig. 3. Uncured 24-ply laminate set up in press with aluminum spacer plates

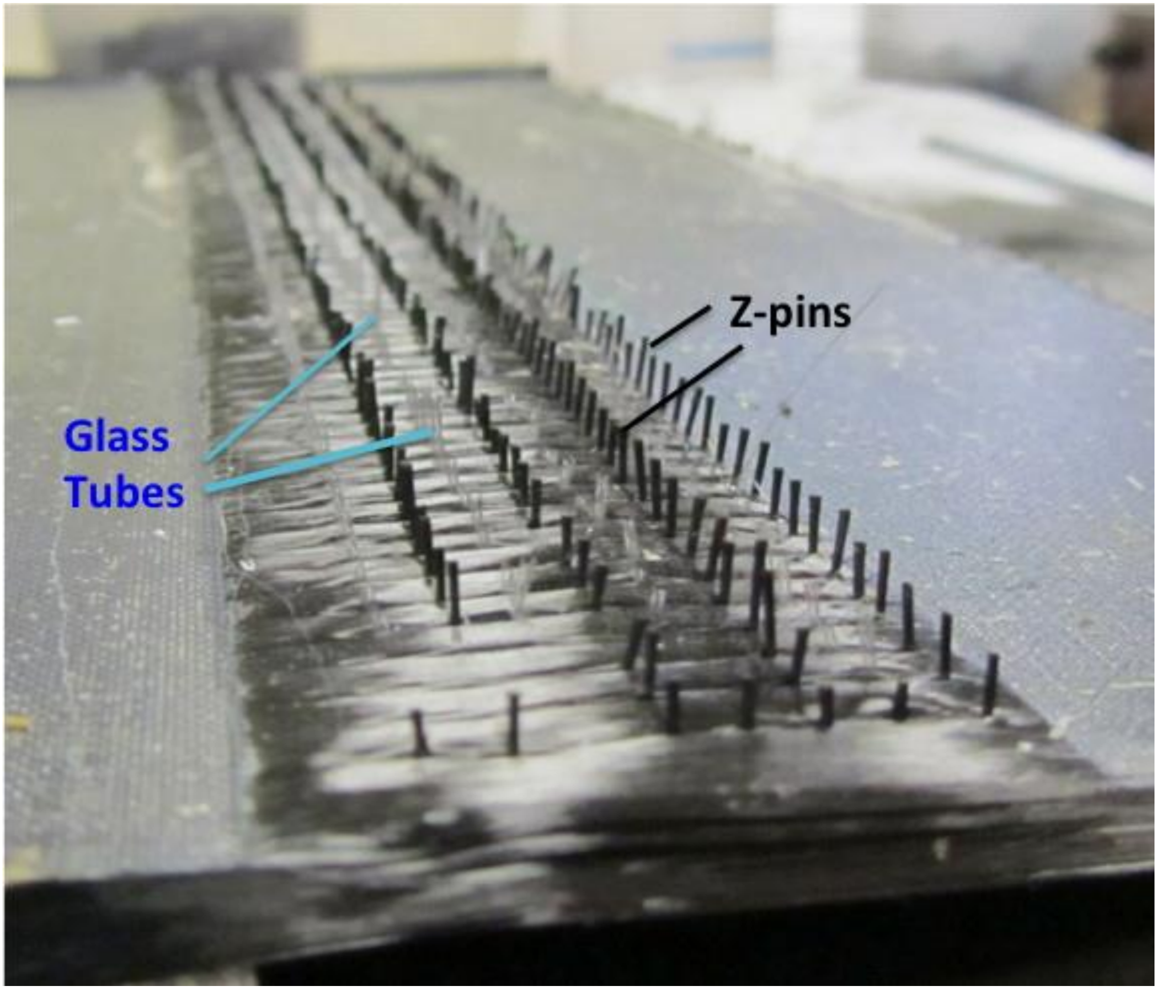


Fig. 4. 24-ply composite panel with inserted Z-pins and hollow glass tubes

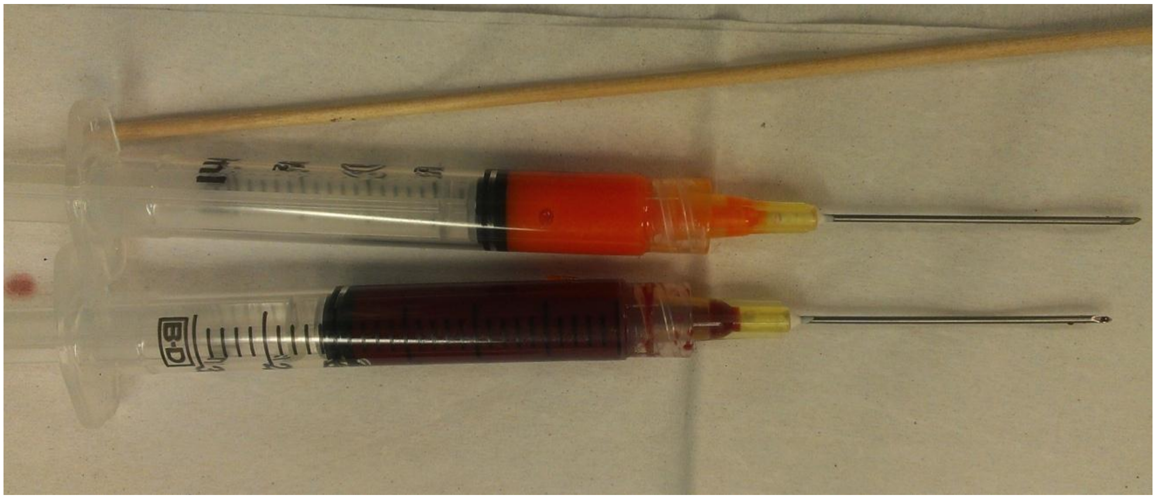


Fig. 5. Self-healing resin and hardener with fluorescent dyes.

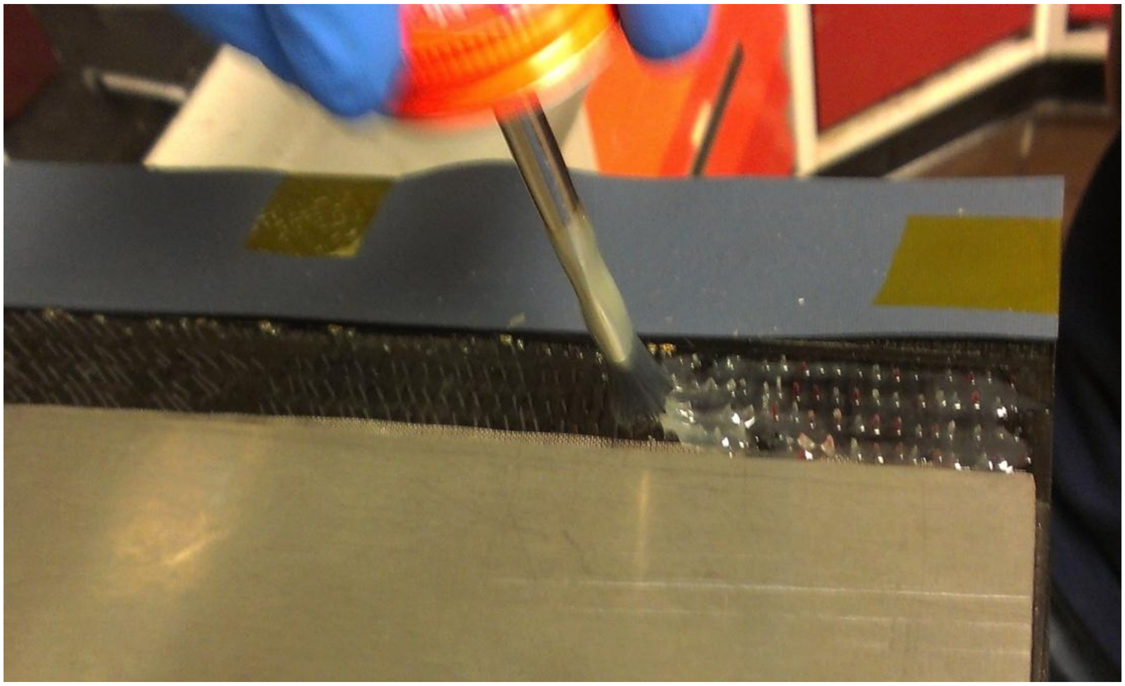


Fig. 6. Rubber cement seals tube ends

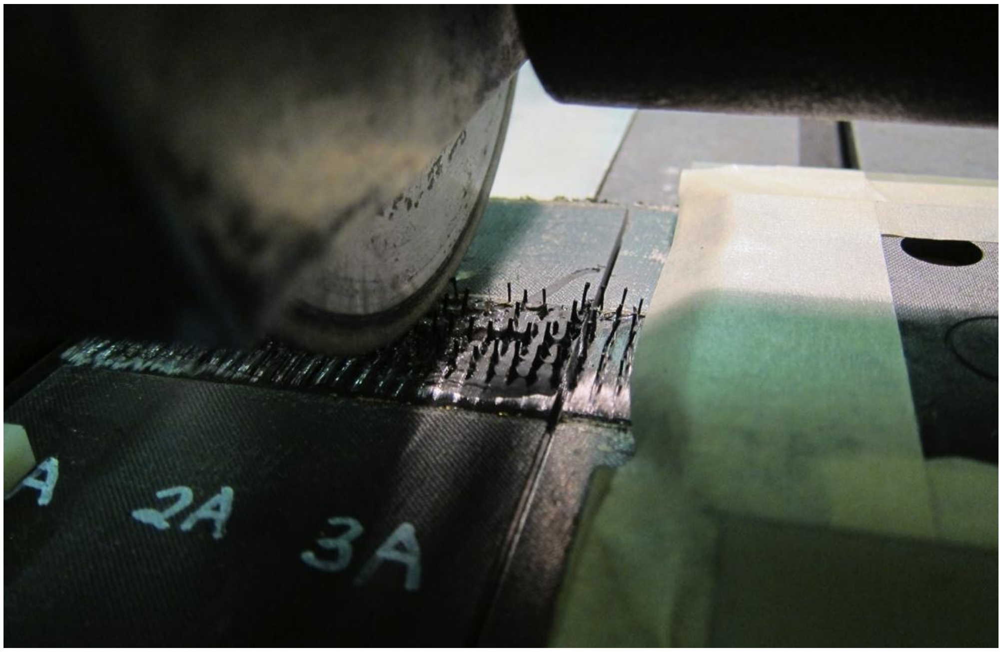


Fig. 7. Diamond saw cutting of 24-ply panel

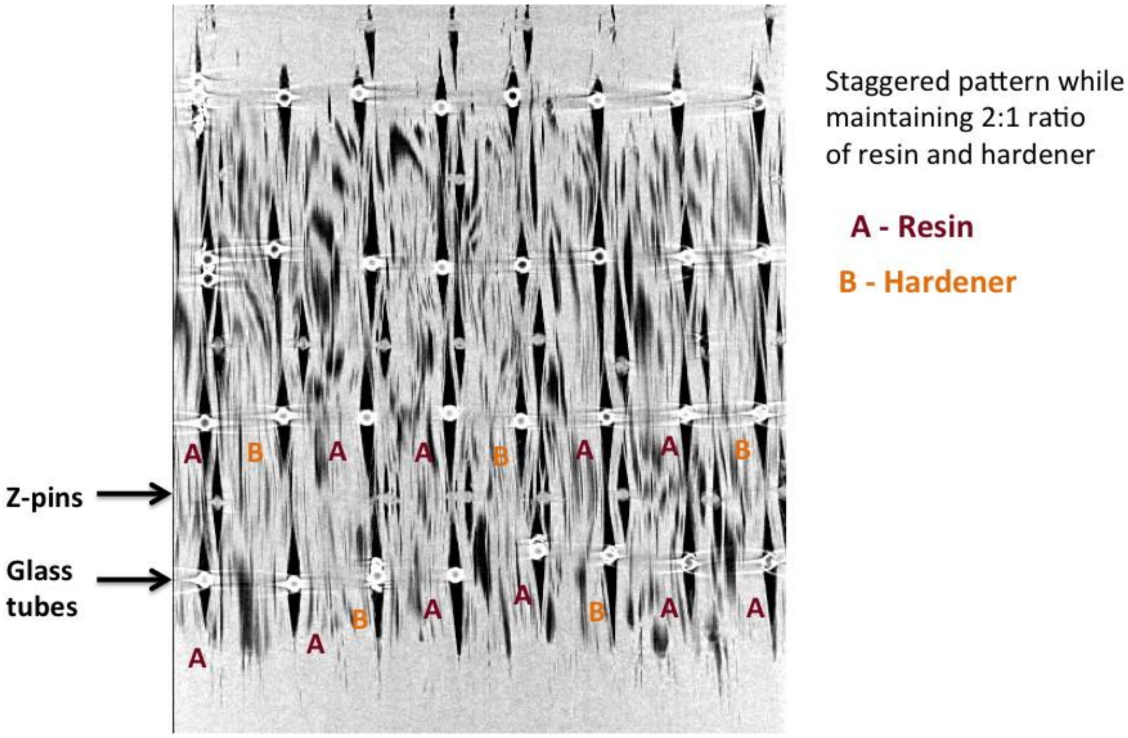


Fig. 8. Illustration of resin and hardener injection pattern on X-ray image of a 24-ply DCB specimen containing inserted Z-pins and hollow glass tubes

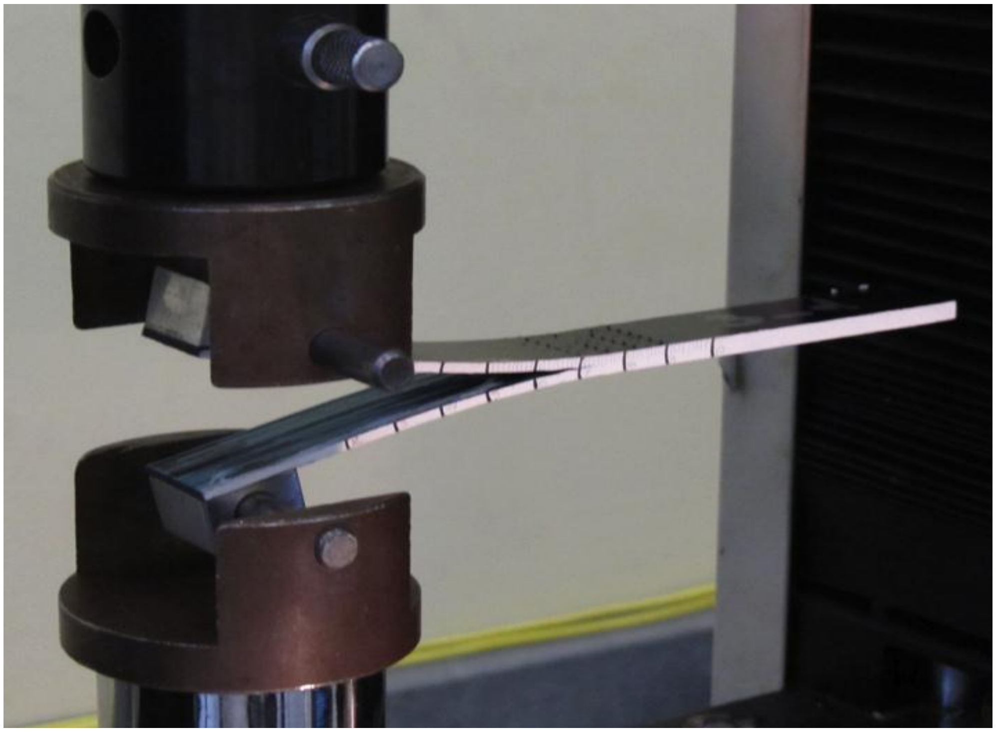


Fig. 9. DCB test of 32-ply specimen

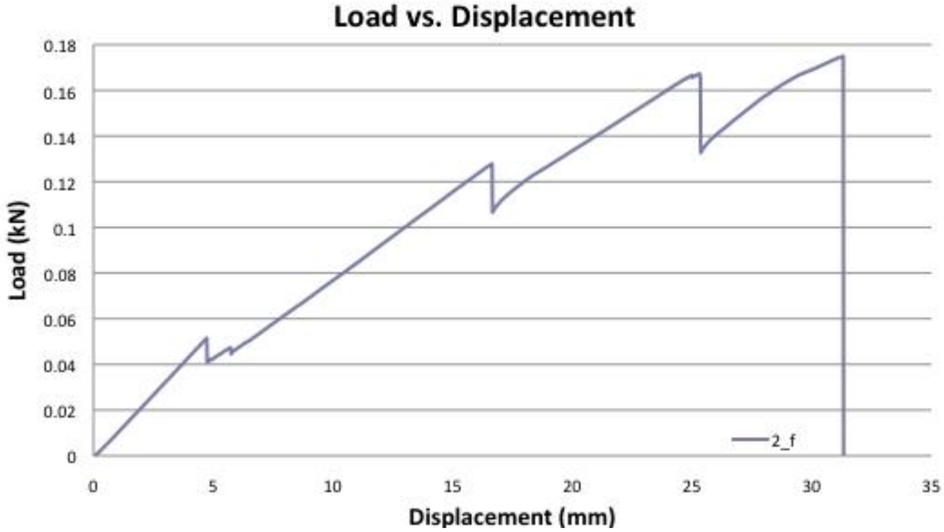
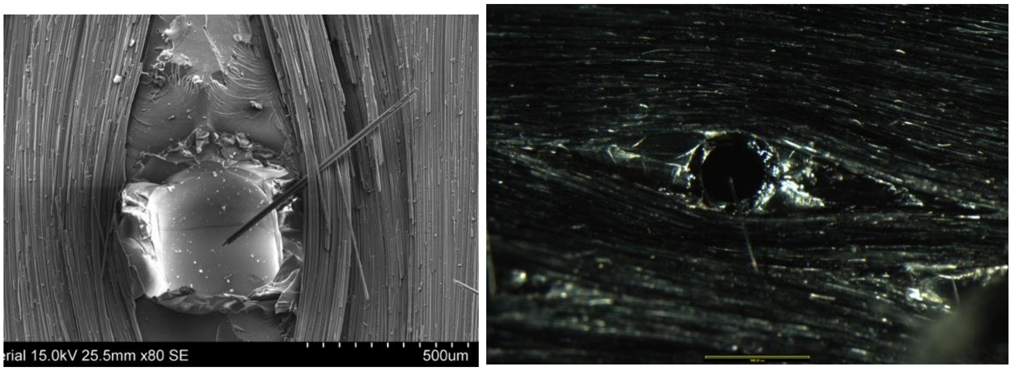
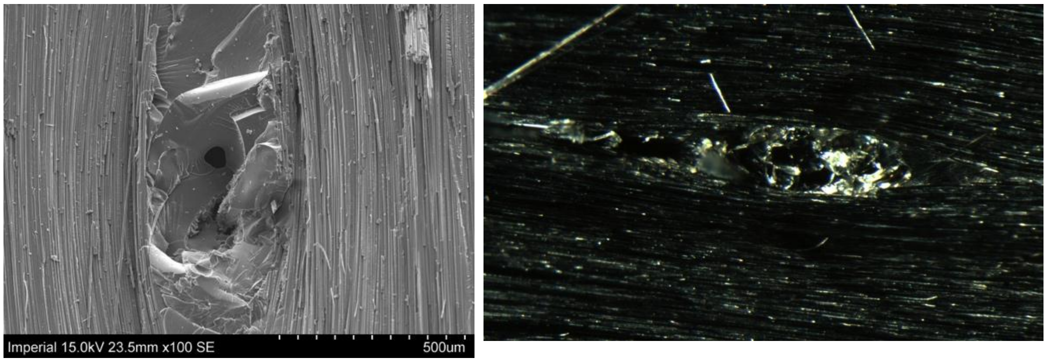


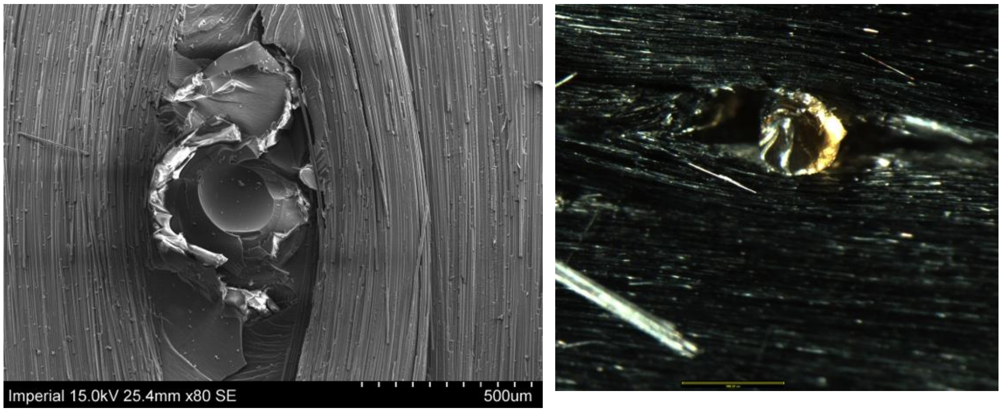
Fig. 10. Typical load-displacement plot for 32-ply laminate DCB test



(a) Broken hollow tube



(b) Crushed tube



(c) Broken tube filled with resin

Fig. 11. Scanning electron and optical microscopy images of fracture plane of 32-ply DCB specimens

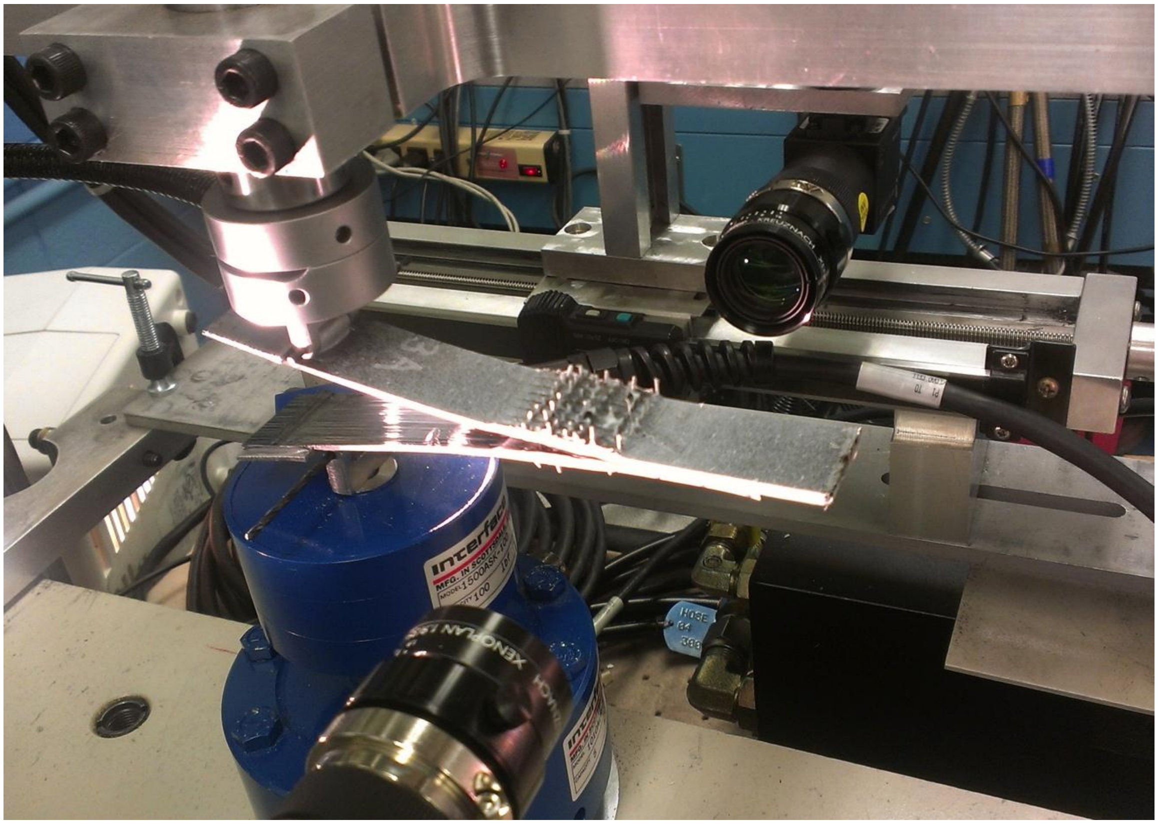


Fig. 12. DCB test of 24-ply specimen 3A showing delamination growing past 4 rows of glass tubes

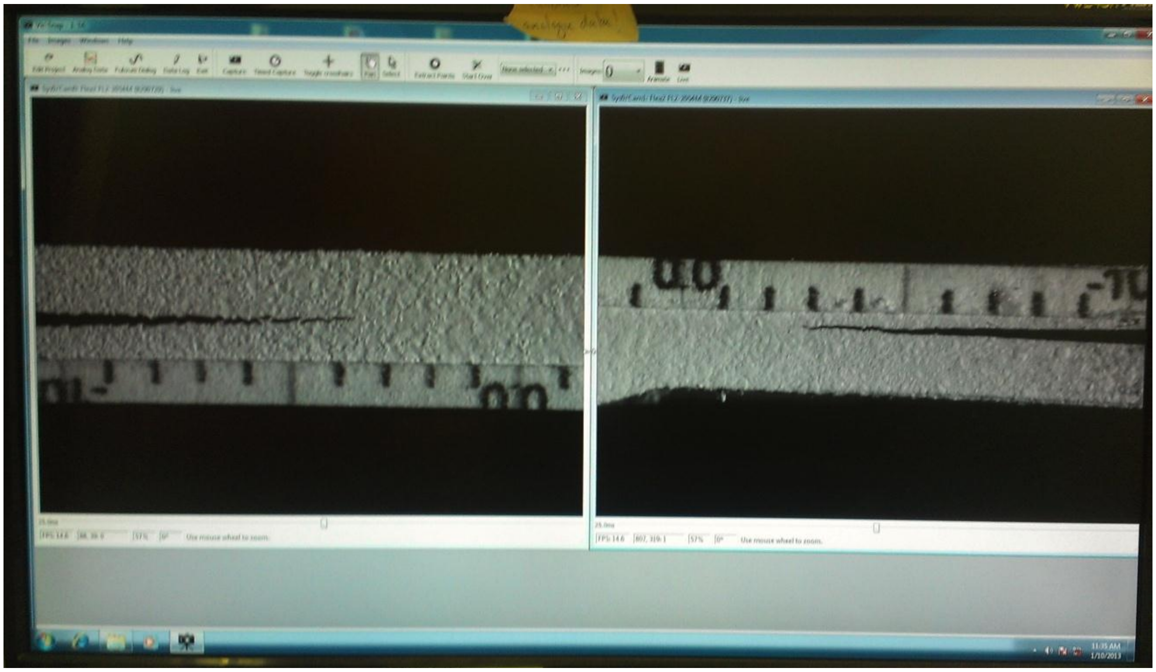


Fig. 13. Video images of delamination growth on edges of 24-ply DCB specimen

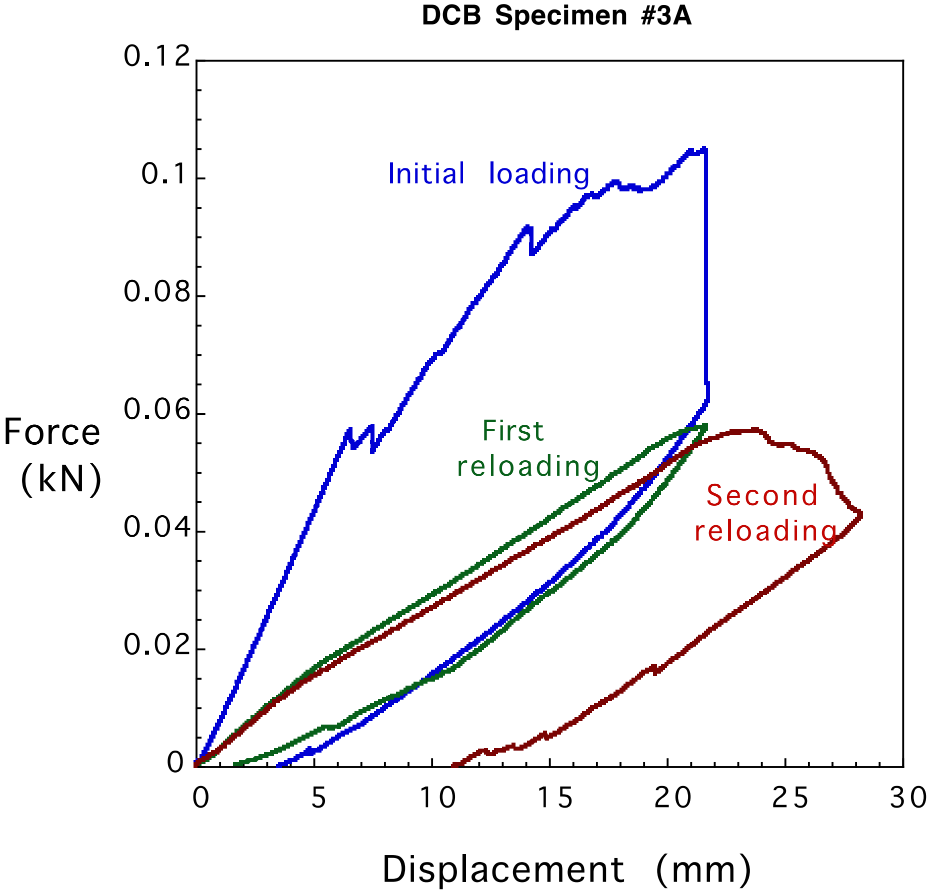


Fig. 14. Load-displacement plots for first 24-ply DCB test, specimen 3A

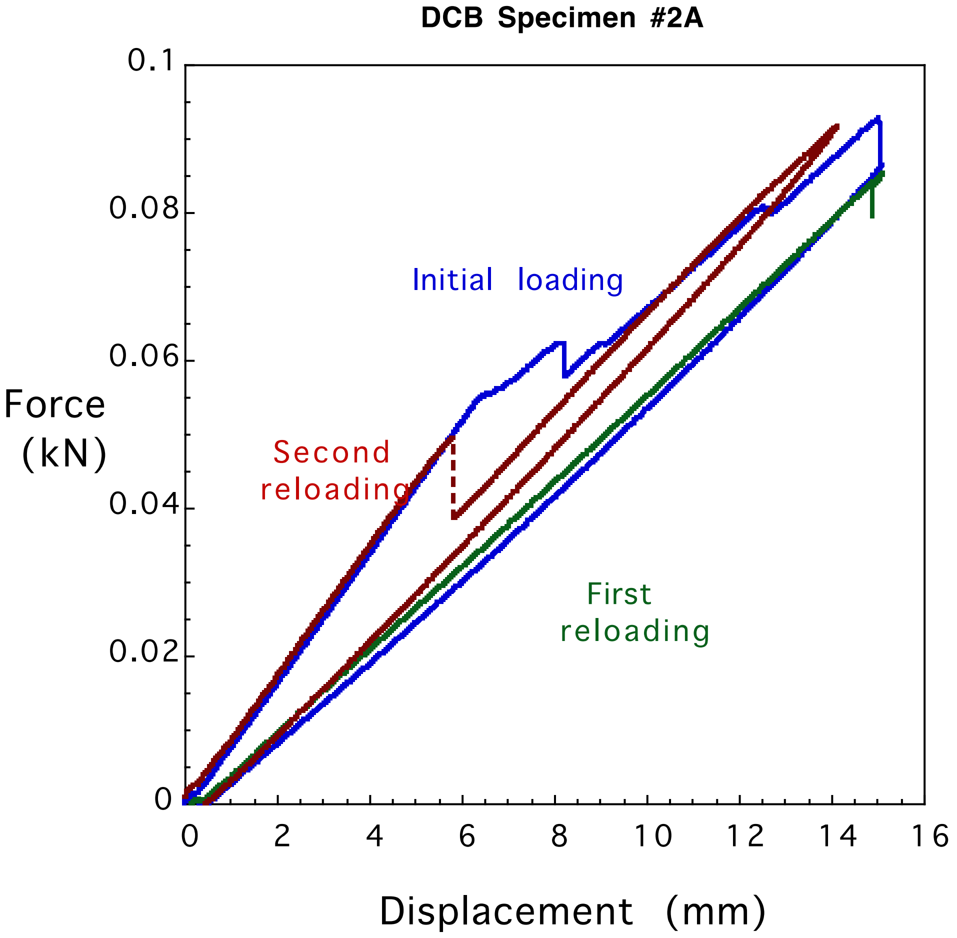


Fig. 15. Load-displacement plots for second 24-ply DCB test, specimen 2A

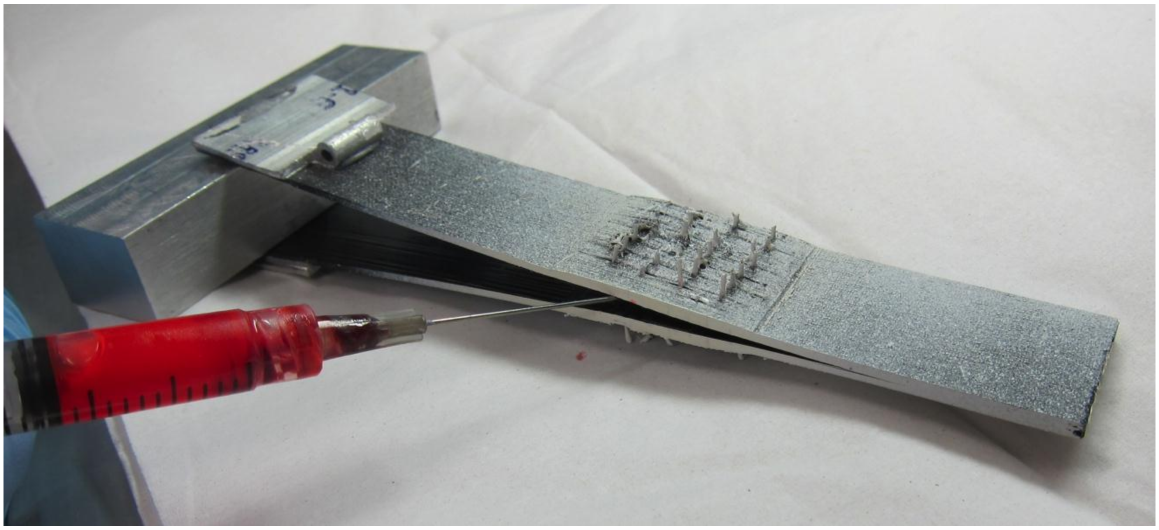


Fig. 16. Injection of premixed resin, hardener and dye into fracture plane

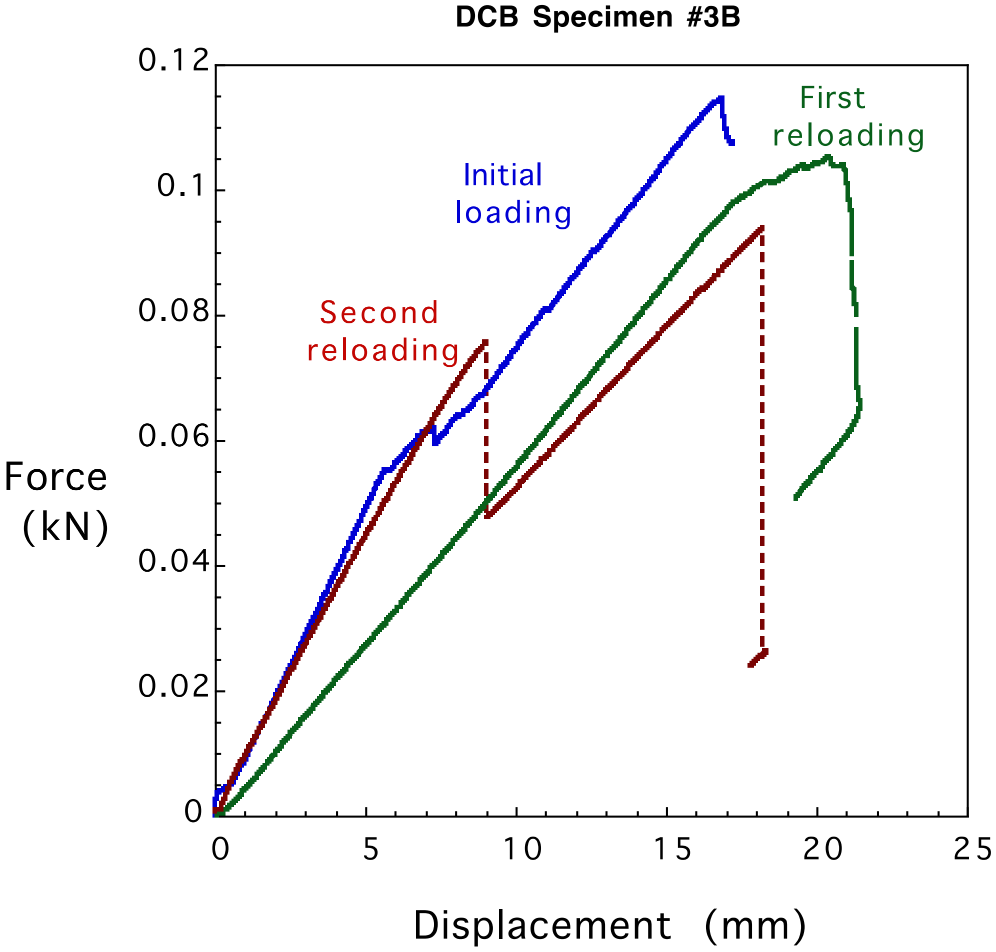


Fig. 17. Load-displacement plots for third 24-ply DCB test, specimen 3B

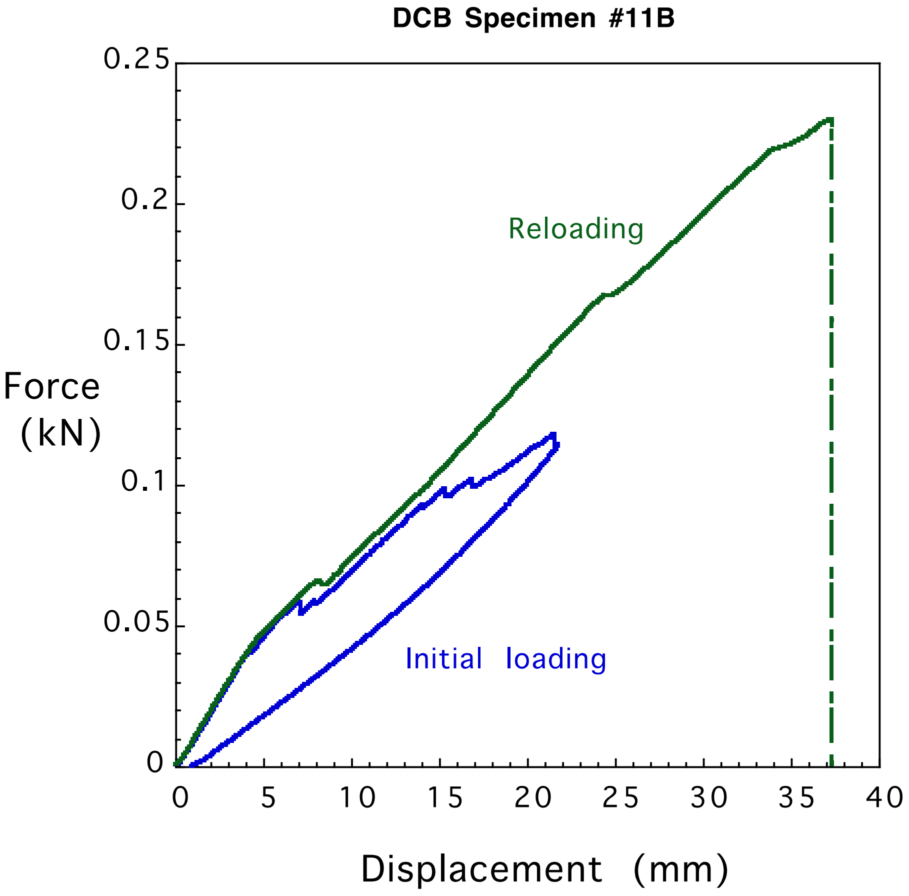


Fig. 18. Load-displacement plots for fourth 24-ply DCB test, specimen 11B

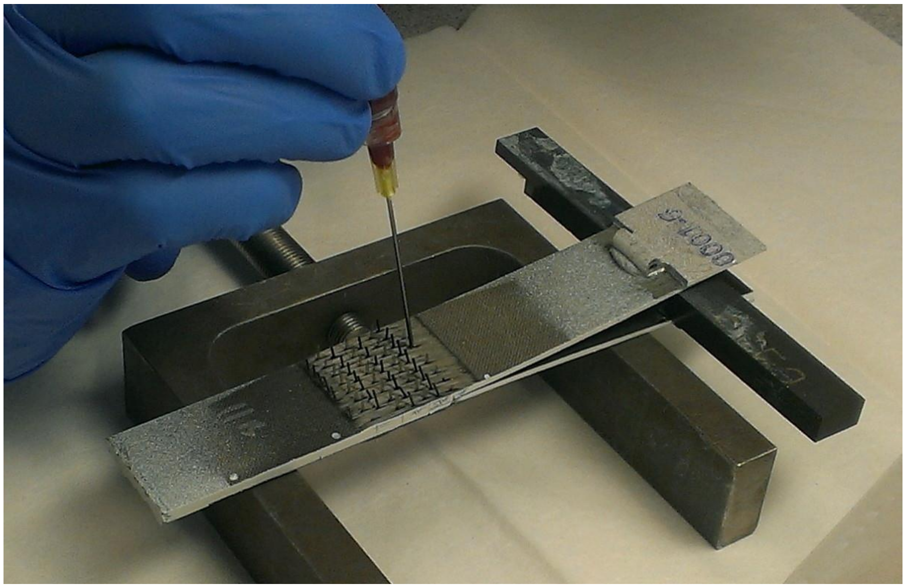


Fig. 19. Injection of premixed resin, hardener and dye into glass tubes

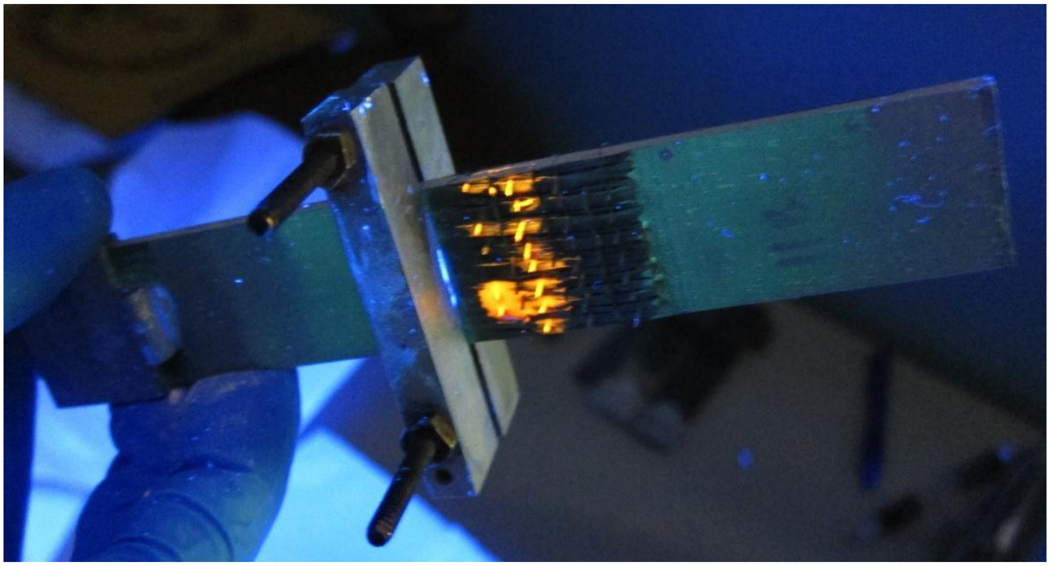


Fig. 20. Ultraviolet surface image of specimen 11B

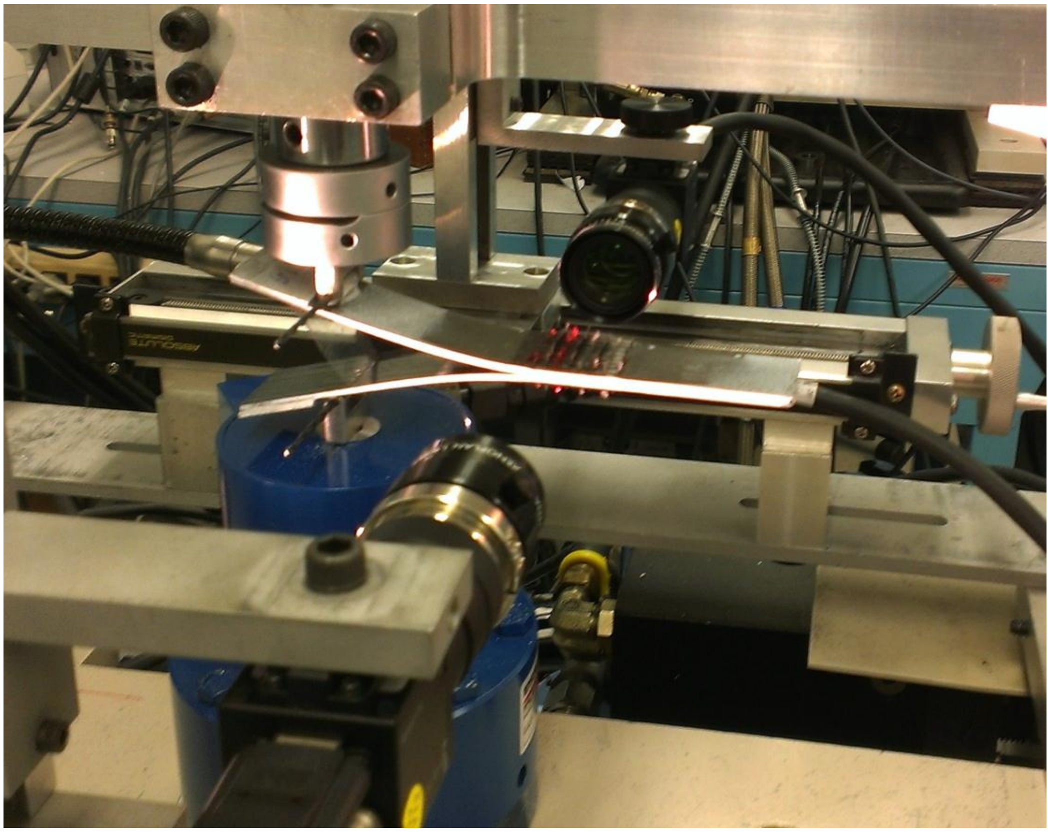


Fig. 21. DCB reloading test showing healing agent in glass tubes

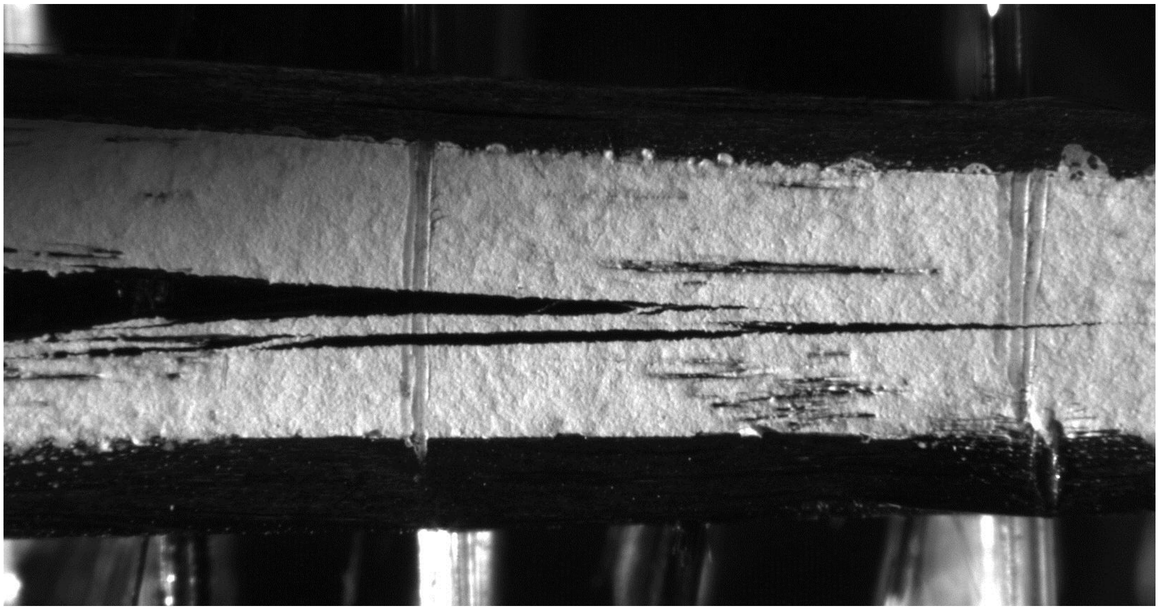


Fig. 22. Formation of secondary branch delaminations

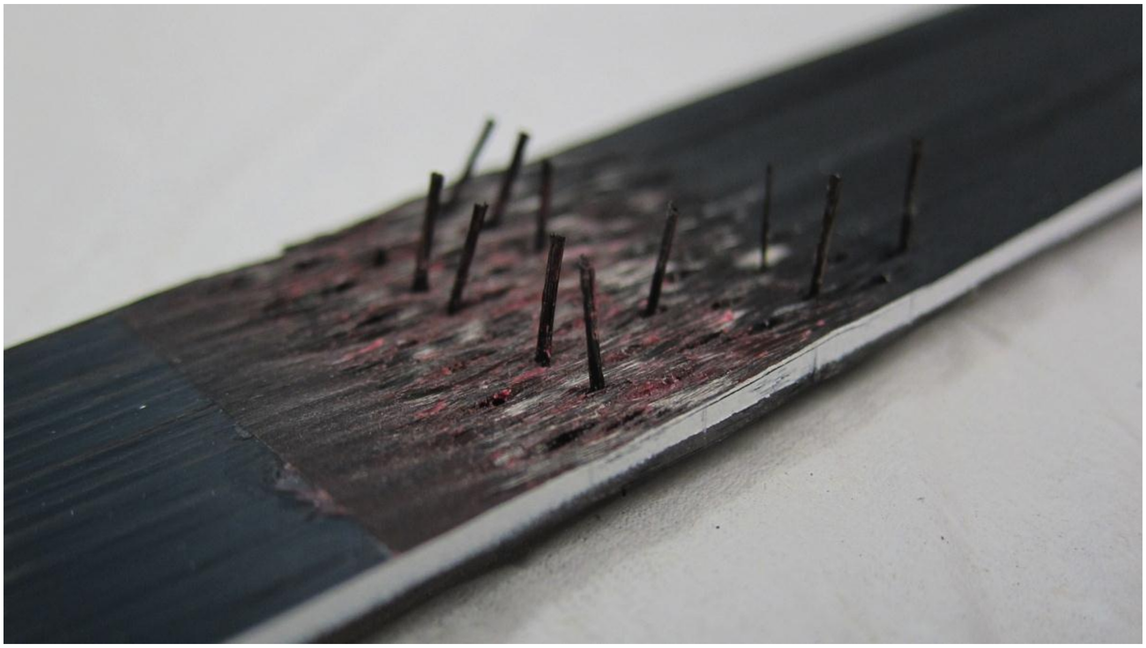


Fig. 23. Fracture surface of specimen 11B indicating the presence of self-healing resin on fracture plane and carbon Z-pins

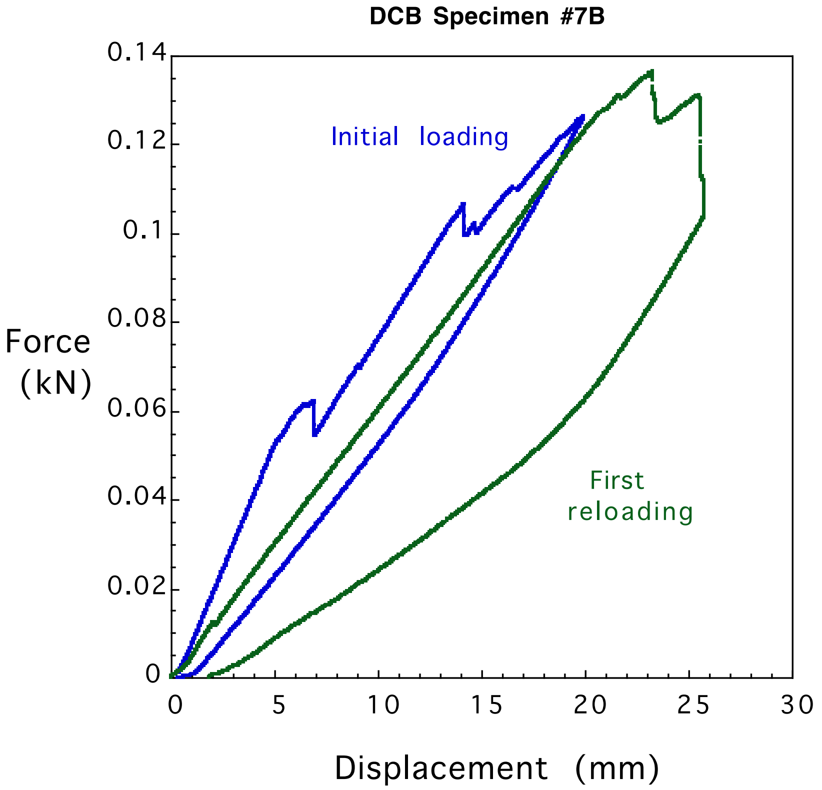


Fig. 24. Initial and first reloading load-displacement plots for specimen 7B

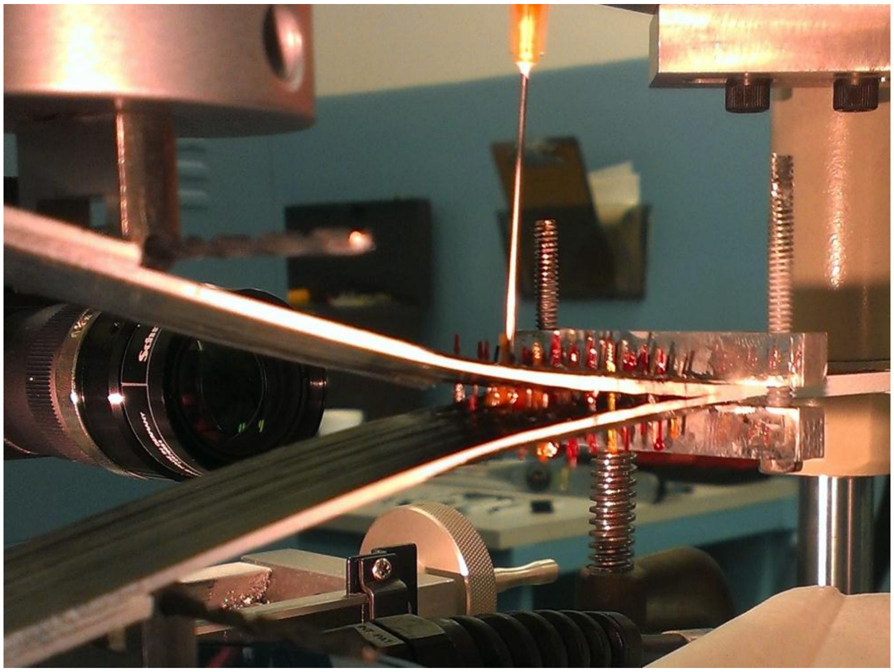


Fig. 25. In-situ injection of resin and hardener in specimen 7B showing droplets in delamination plane

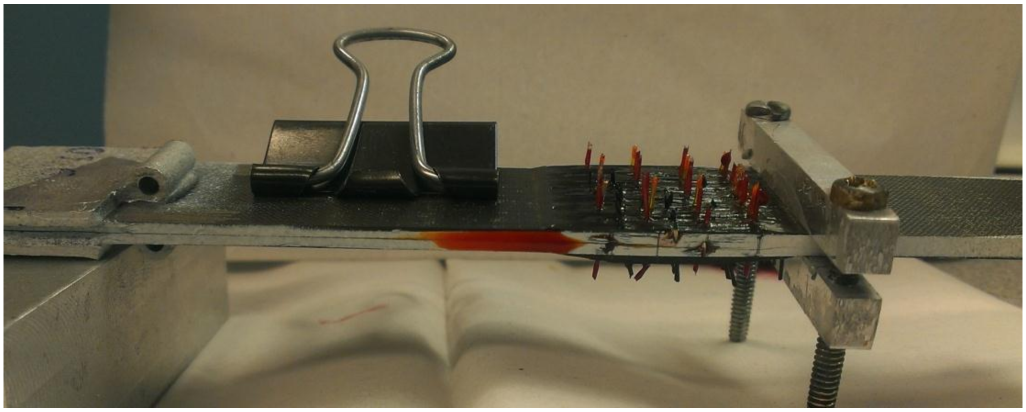


Fig. 26. Sealing resin visible on edge of specimen 7B

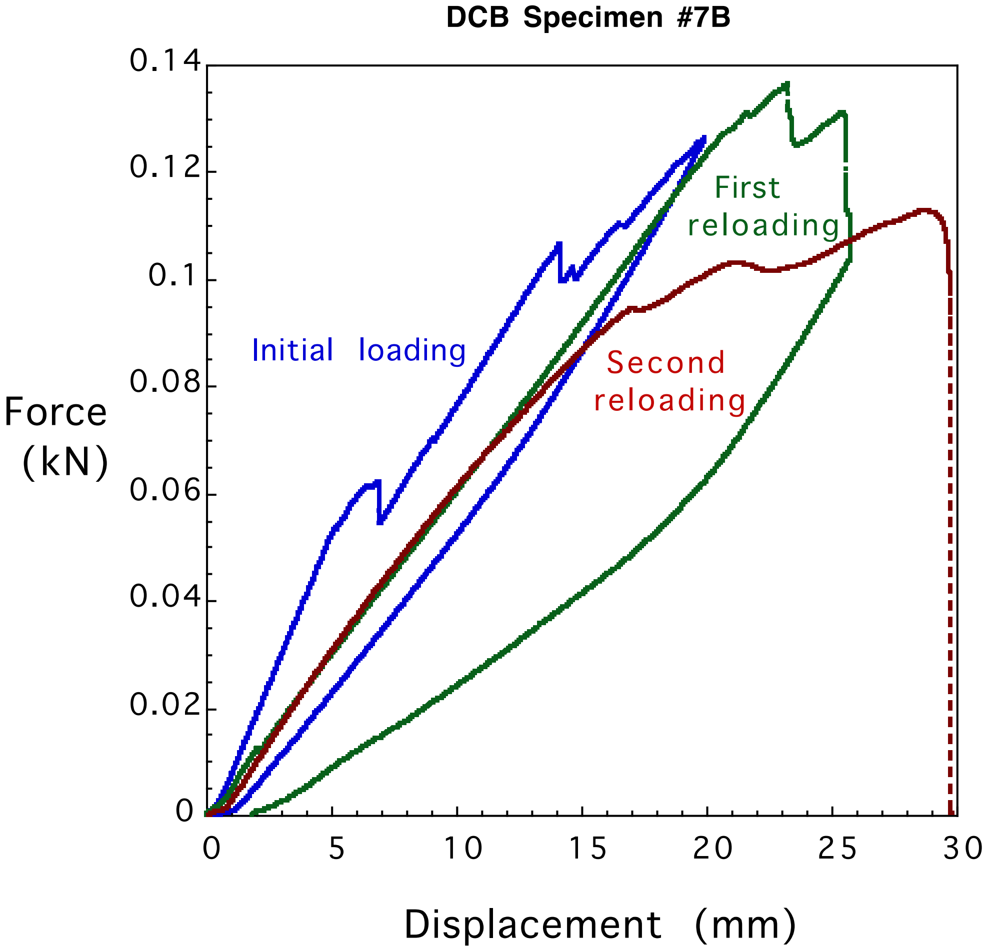


Fig. 27. Load-displacement plots for specimen 7B

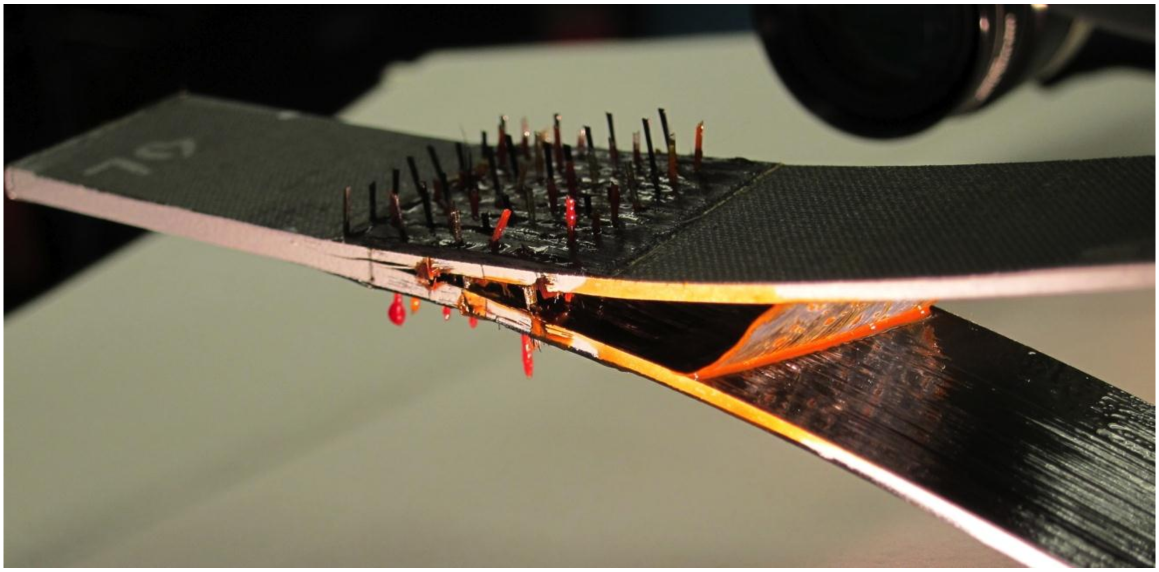


Fig. 28. Second reloading DCB test of Specimen 7B showing resin leakage

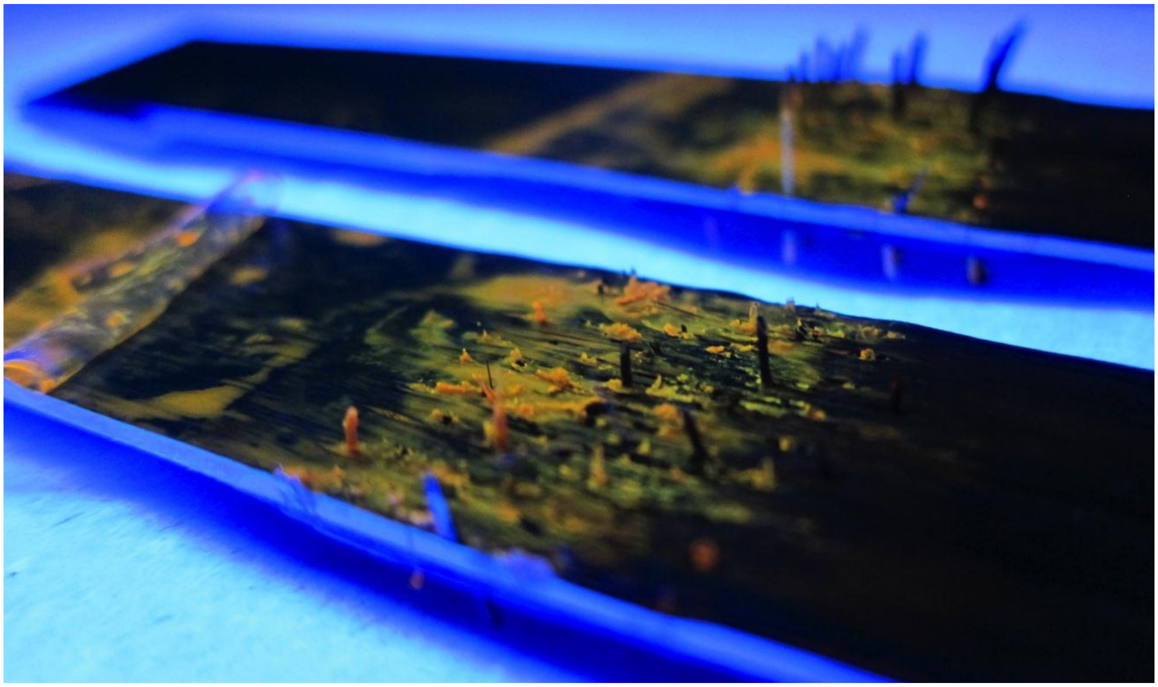


Fig. 29. Ultraviolet light image of fracture plane for specimen 7B

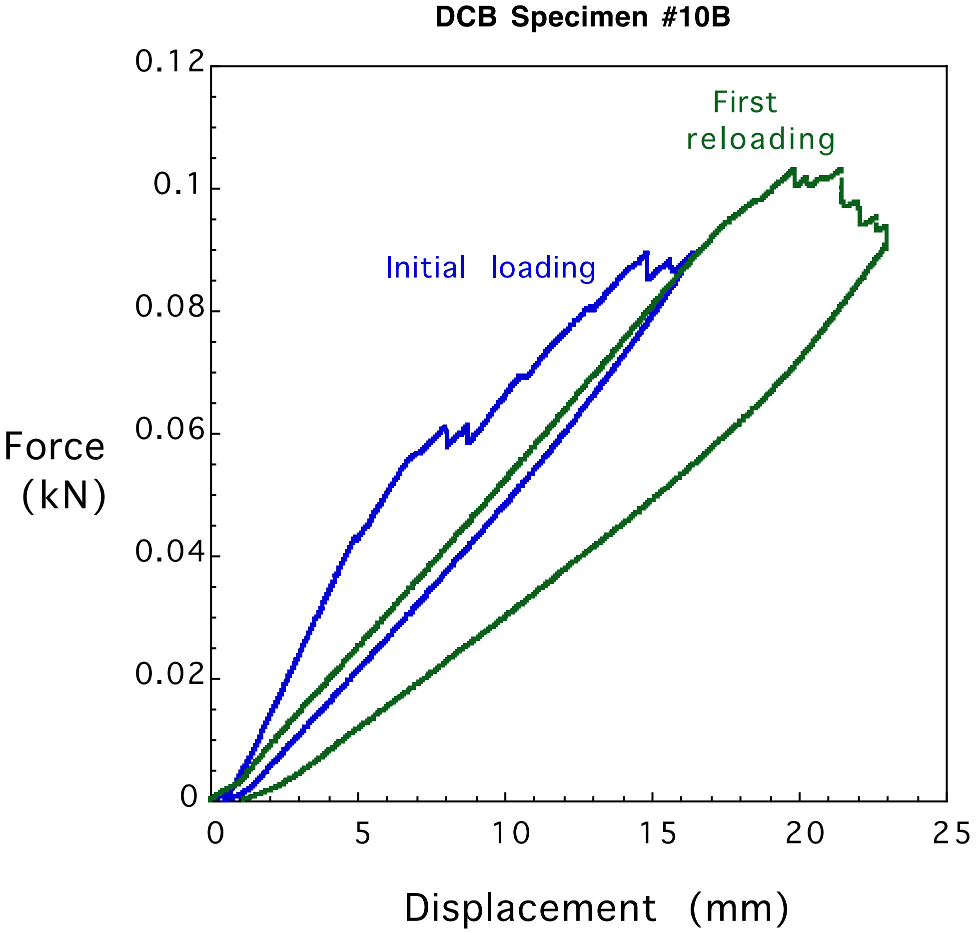


Fig. 30. Initial and first reloading load-displacement plots for specimen 10B

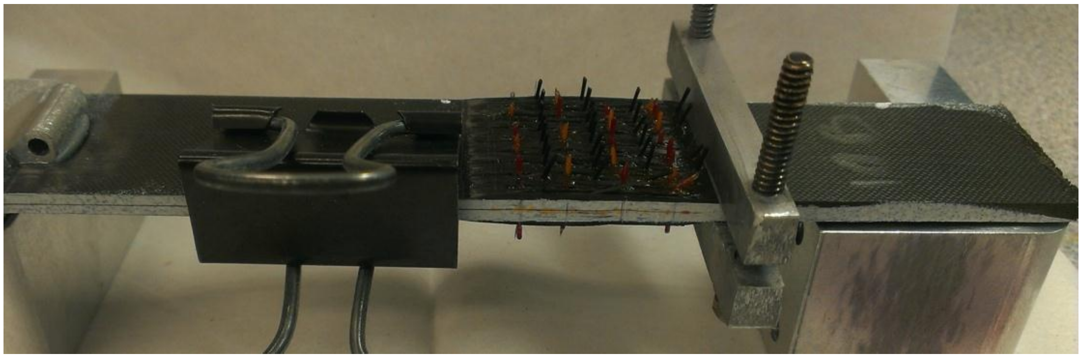


Fig. 31. Sealing resin visible on edge of specimen 10B

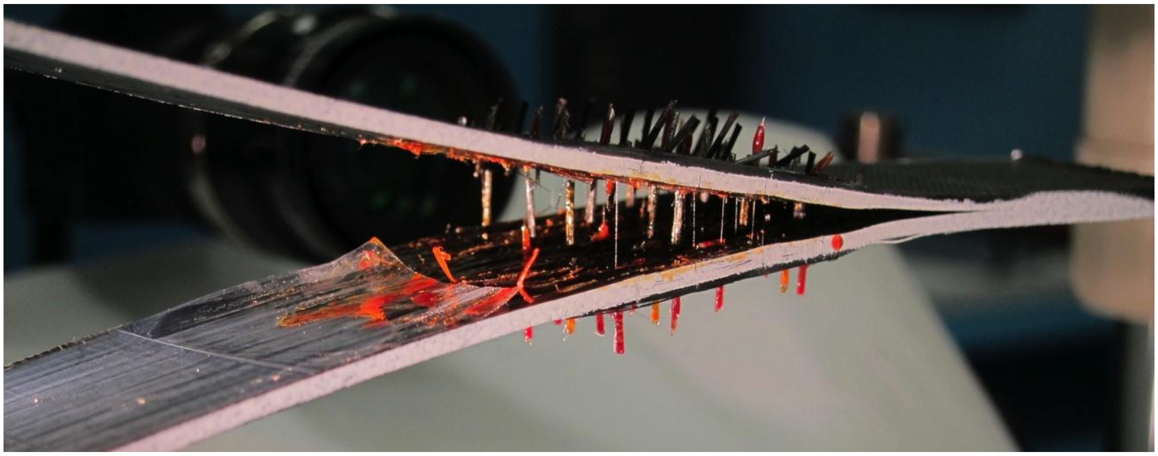


Fig. 32. Second reloading DCB test of Specimen 10B

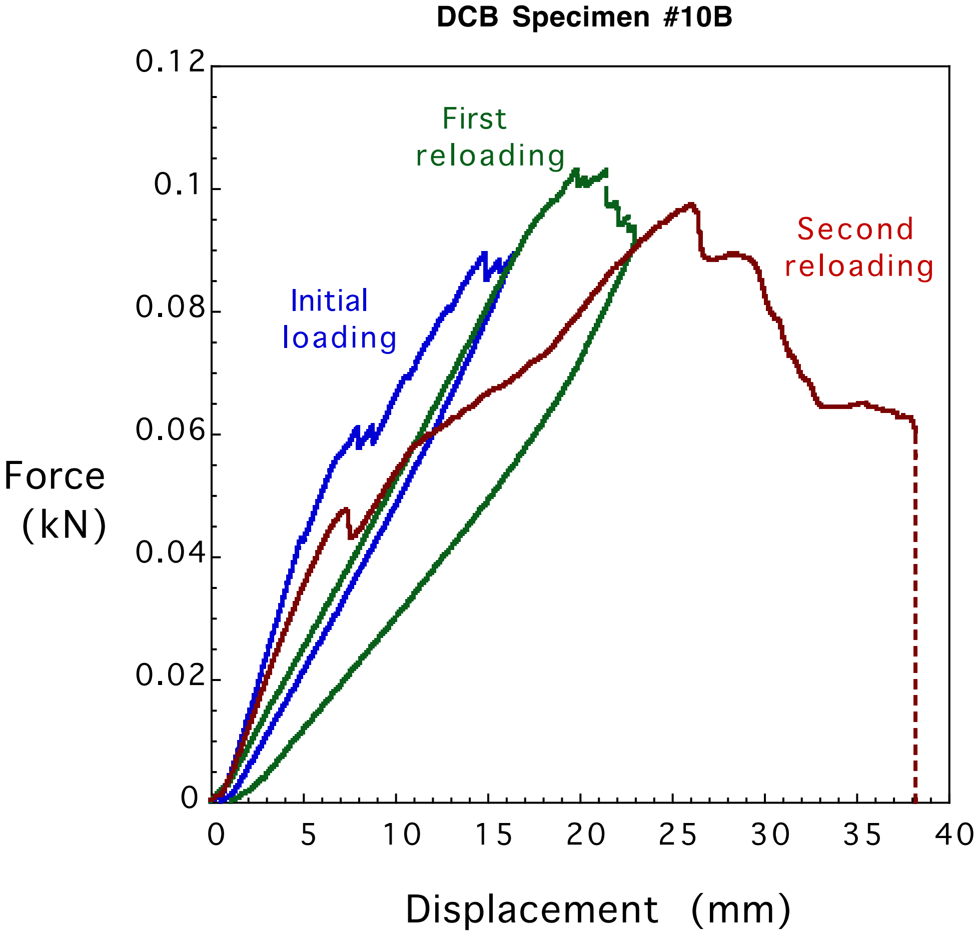


Fig. 33. Load-displacement plots for specimen 10B

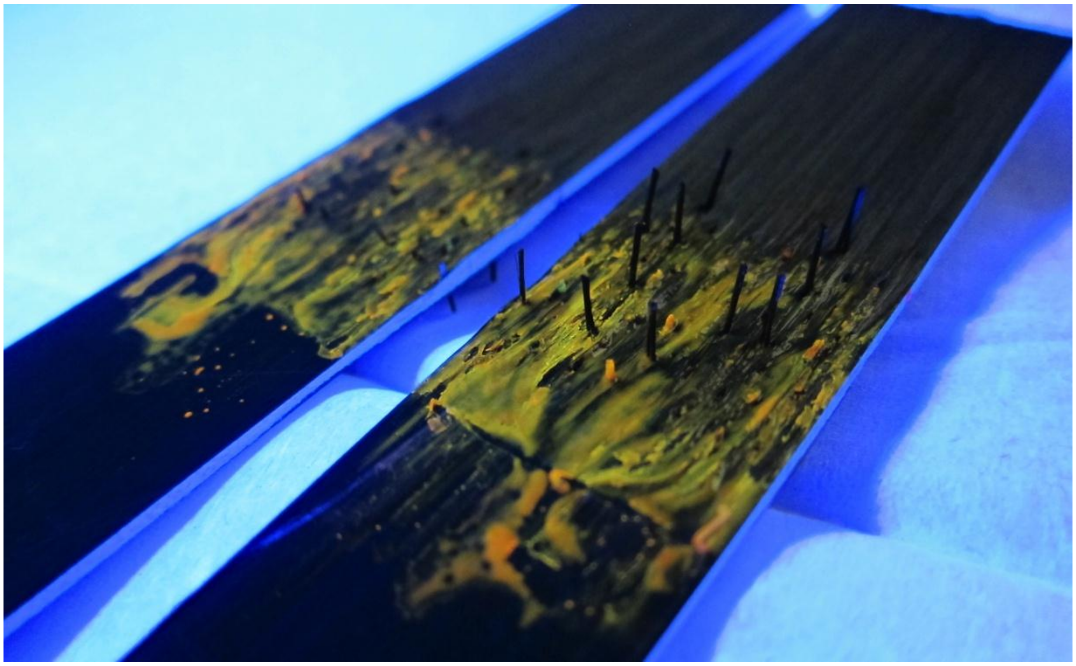


Fig. 34. Ultraviolet light image of fracture plane for specimen 10B

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14. ABSTRACT

A study was undertaken to develop a prototype method for adding through-thickness hollow glass tubes infused with uncured resin and hardener in a carbon Z-pin through-thickness reinforcement field embedded in a composite laminate. Two types of tube insertion techniques were attempted in an effort to ensure the glass tubes survived the panel manufacturing process. A self-healing resin was chosen with a very low viscosity, two component, liquid epoxy resin system designed to be mixed at a 2-to-1 ratio of epoxy to hardener. IM7/8552 carbon epoxy double cantilever beam (DCB) specimens were cut from the hybrid Z-pin and glass tube reinforced panels and tested. In-situ injection of resin and hardener directly into glass tubes, in a staggered pattern to allow for 2-to-1 ratio mixing, resulted in partial healing of the fracture plane, but only if the injection was performed while the specimen was held at maximum load after initial fracture. Hence, there is some potential for healing delamination via resin and hardener delivered through a network of through-thickness glass tubes, but only if the tubes are connected to a reservoir where additional material may be injected as needed.

15. SUBJECT TERMS
Delamination; Self-Healing; Z-PIN

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