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**MECHANICAL CHARACTERIZATION OF TWO THERMOPLASTIC
COMPOSITES FABRICATED BY AUTOMATED TOW PLACEMENT**

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ABSTRACT

AS4/PEEK towpreg and IM7/Radel 8320 slit tape were used to make flat panels by automated tow placement. The panels were tested in notched and un-notched tension, notched and un-notched compression and compression after impact (CAI) at room temperature and under hot/wet conditions (notched and un-notched compression and CAI only). The properties were compared with AS4/PEEK tape laminate properties found in the literature. The tow placed AS4/PEEK material was stronger in tension but weaker in compression than the AS4/PEEK tape laminates. The tow placed AS4/PEEK was stronger but less stiff than the tow placed IM7/Radel 8320 in all compression tests. The IM7/Radel performed better in all other mechanical tests. The IM7/Radel outperformed the AS4/PEEK in all CAI tests.



INTRODUCTION

Advanced polymeric composite materials offer significant potential for weight savings and performance advantages over traditional aircraft materials. A major goal of the NASA Advanced Composite Technology (ACT) program is to develop these materials for use in primary structure of commercial aircraft. Compared to metallic materials, composites offer tailorability of properties along with very high specific strength and stiffness. However, laminated composite structures are expensive to manufacture and less damage tolerant than desired. If advanced composites are to be used in the primary structure of commercial aircraft, the problems of high cost and low damage tolerance must be overcome.

When first introduced, thermoplastic composites such as graphite/PEEK were heralded as offering a solution to the low damage tolerance of traditional graphite/epoxy laminates. Although the improved toughness of thermoplastic matrix composites is well documented, their high cost relative to traditional materials has prevented their widespread use in aircraft structures.

Much effort has been made to reduce the cost of manufacturing composite structure by using processes such as filament winding and automated tow placement. While the filament winding process has been utilized in the aerospace industry for some time, it is generally restricted to certain volumetric shapes and cannot manufacture a concave form. A specialized form of filament winding called automated tow placement (ATP) or fiber placement is capable of manufacturing simple volumetric shapes, flat panels and complex (including concave) shapes. The process uses a multi-axis robotic machine (ref. 1) to lay down multiple tows as a band, forming a laminated structure (see figure 1). The band location and angles are precisely controlled with material cut, add and compaction features incorporated into the process. ATP has been identified as a cost effective automated manufacturing process capable of producing the large complex shapes needed for the next generation of commercial aircraft (ref. 1 and 2). While demonstrating cost saving potential, most ATP work has been done with thermoset materials, which often have poor damage

tolerance. There is little published data for tow placed thermoplastics.

The objective of this work was to evaluate the potential of using cost effective ATP in conjunction with damage resistant thermoplastic materials to fabricate composites for use as primary aircraft structure. The approach was to select two graphite/thermoplastic (Gr/Tp) material systems, fabricate flat panels by ATP and perform standard tests to evaluate the mechanical properties. The ATP material properties were also compared to the properties of tape laminates. The mechanical property characterization included notched and un-notched tension and compression, compression after impact, and environmental degradation under hot/wet (H/W) conditions.

MATERIALS AND PROCESSING

The thermoplastic material systems evaluated in this study were AS4/PEEK towpreg and IM7/Radel-8320 (formerly Radel-X) slit tape. The two materials were selected from several which were available from suppliers and capable of being tow placed. AS4/PEEK was selected so that comparisons could be made to existing data on AS4/PEEK prepreg tape material. The IM7/Radel material offered somewhat different constituent material properties and processing temperatures than the AS4/PEEK. Tables 1 and 2 show the fiber and resin properties for the two material systems (ref. 3-6).

Under contract to NASA Langley, Hercules Aerospace Company, Composite Products Group used automated tow placement to manufacture the flat composite panels with the above materials. A modest development effort was made to "auto-consolidate," or "consolidate on the fly." In an auto-consolidation process, the thermoplastic material is heated to its processing temperature and consolidated in-situ with a compaction roller. Early efforts involved making small hoops or cylinders with a filament winding machine. The development work was done with AS4/PEEK. A special fiber placement head was developed to apply heat as the tows were laid down on the tool or part. A Sylvania hot air flameless

torch was selected as the heat source. The manufacture of quality (low void content) filament wound rings was found to be fairly easy. However, the manufacture of the flat panels was found to be quite difficult. Two significant problems were identified.

First ply adhesion was found to be very difficult in the tow placement of flat panels. The first layer of the composite must adhere to the tool or mandrel so that subsequent plies can be added successfully. The material must adhere to the tool at high processing temperatures and yet easily release from the tool once processing is complete. This problem is less significant when "tacky" thermosets are used or when the first ply can be completely wrapped under tension around a volume of revolution. To overcome the problem of first ply adhesion in the flat panels of this study, the panels were manufactured oversized (see figure 2). An AS4/PEEK bi-direction tape (woven material) was mechanically fastened to the flat tool outside the perimeter of the required panel area. During the lay-down of the first layer, the beginning and end of each pass adhered to the bi-directional tape. Once the second ply was placed the first ply seated down in the correct position with minimal gaps or overlaps. Once all the layers were laid down, the excess material was then trimmed away leaving a panel of the required size.

The second major difficulty encountered in auto-consolidation of the flat panels was related to the difference between tow placement and filament winding processes. The development work was done with a filament winding machine. However, automated tow placement was used to make the flat panels. In the filament winding of thermoplastic materials, the winding head stays in contact with the mandrel or composite part making consolidation "on the fly" simpler. Reference 7 discusses process development of in-situ consolidation of ring-type specimens and cylinders with filament winding. In tow placement the fiber placement head accelerates from a still position, traverses a set distance and then decelerates to a complete stop where the tow is cut. The head is then lifted from the surface of the panel and indexed to another position. The unavoidable variation in material lay-down rate and discontinuity of contact between the placement head and composite part creates a complicated process control problem for in-situ consolidation. The AS4/PEEK towpreg was fairly insensitive to this variation. However the IM7/Radel was very sensitive to the process

parameters; hence, the extent of in-situ consolidation was not constant across any given lay-down pass. Only 40% consolidation (based on visual observation and past experience by the contractor) was achieved near the beginning and end of each pass while around 75% consolidation was accomplished near the middle of each pass. To achieve full consolidation on the fly specific attention needs to be given to controlling the heat source as a function of machine speed (open-loop feedback) or material temperature (closed loop feedback).

Due to the complexity of auto-consolidation with tow placement, full consolidation was not achieved "on the fly" at any lay-down rate. However, as auto-consolidation was not the goal of the research, the processing was completed by vacuum bag/autoclave cycles in a manner similar to that used for tow placed thermoset parts. Autoclave pressure was 200 psi for both material systems. The maximum processing temperatures were 720°F and 650°F for the AS4/PEEK and the IM7/Radel, respectively. Ultrasonic testing was conducted by Hercules to insure quality and determine the extent of consolidation. The panels were reprocessed in the autoclave several times until nearly void free.

The flat panels were made with a quasi-isotropic lay-up, [45/0/-45/90]_{3s} for un-notched tension and [45/0/-45/90]_{6s} for compression and notched tension. Completed panels were again ultrasonically tested at NASA Langley to verify quality, void free panels.

TEST METHODS AND PROCEDURES

Tension, compression, 90° compression, notched tension, notched compression and compression after impact (CAI) testing was performed with the quasi-isotropic flat panels. Specimen configurations are shown in figure 3. Test procedures as described in reference 8 were followed unless otherwise stated. Three specimens were tested for each configuration with the exceptions of 90° compression and room temperature CAI where two

specimens (each) were tested. Specimens were instrumented with 350 ohm back-to-back strain gages as shown in figure 3. Data were gathered with a 16-bit resolution A/D micro-computer-based data acquisition system. Tension tests (notched and un-notched) were performed in a 50 kip electro-hydraulic test machine equipped with hydraulic grips at a constant stroke of 0.05 in./min. All compression tests were performed in a 120 kip hydraulic test machine at a constant rate of 0.05 in./min.

Tension specimens were un-tabbed, but each end had a coarse grit paper and Lexan film between the knurled grips and specimen. The un-notched compression test utilized a specimen configuration and fixture developed at NASA Langley to evaluate the compression properties of composites. The test is referred to as short block compression (SBC). The SBC fixture, shown in figure 4, applies end loading while preventing a brooming type failure. The CAI test specimens were impacted with a low-velocity air gun apparatus which fires a 1/2-in. diameter aluminum ball (0.0065 lb mass). An impact energy of 1500 in.-lb/in. of specimen thickness was used (approximately 550 ft/sec velocity, equalling 30 ft-lb of energy). Impacted panels were loaded in a special fixture, shown in figure 5. The fixture utilizes clamped ends and simple support knife edges on each side. The specimens are end loaded and the clamped ends prevent brooming failures. The impacted specimens were C-scanned after impact, before compression testing to determine the extent of the impact damage. Fiber volume fraction for each large panel was determined by acid digestion of small samples obtained from different locations within each panel (ref. 9).

While the majority of the testing was performed on unconditioned specimens at laboratory conditions, some environmental testing was performed on notched and un-notched compression (three specimens each) and CAI specimens (one specimen). Specimens were immersed in 160°F water for 45 days. The specimens were weighed before and after exposure to determine moisture gain and then instrumented with strain gages. The specimens were then tested at 180°F, usually within one or two hours. This type test is referred to as hot/wet (H/W) testing.

RESULTS AND DISCUSSION

The results of the mechanical testing are presented as averages in tables 3 through 5. The fiber volume fractions ranged from 53.9% to 57.2% for the AS4/PEEK and 63.6% to 64.0% for the IM7/Radel. Normalizing the mechanical properties to a 60% fiber volume fraction did not significantly affect property trends or comparisons; hence, data shown are actual test values. Typical stress-strain plots for each material are shown in figure 6. Moduli were calculated by a linear regression over the range of 0.1% to 0.3% strain to avoid initial loading artifacts.

Tension Properties

The quasi-isotropic tow placed AS4/PEEK and IM7/Radel tensile properties are shown in figure 7 along with quasi-isotropic AS4/PEEK tape (prepreg) laminate data from reference 10. The bars represent the average values listed in the tables and the lines drawn through the bars indicate the full range of the repeat tests. The tow placed AS4/PEEK composite was slightly stronger than the AS4/PEEK tape composite (10%), but both possessed similar moduli. It should be noted that the tension testing of the tape material in reference 10 was performed with a 48 ply quasi-isotropic material while the tension testing of the tow placed material was performed with a similar layup of only 24 plies.

Comparing the two tow placed materials, the IM7/Radel had an approximately 32% greater un-notched tensile strength and modulus than the AS4/PEEK. This fact is not surprising in that tension properties are dominated by fiber behavior. IM7 fiber has a significantly greater strength (30%) and modulus (21%) than AS4 fiber (see table 1). The two composite systems exhibited similar tensile failure strains and in-plane Poisson's ratios (tables 3 and 4). Both the AS4/PEEK and IM7/Radel exhibited approximately 50% lower

tensile strength when tested with a 0.25-in. diameter hole.

Compression Properties

Figure 8 illustrates the room temperature and hot/wet compression properties of the quasi-isotropic tow placed AS4/PEEK and IM7/Radel composite materials. Again, the bars represent the average values and the lines drawn through the bars indicate the full range of the repeat tests. Also shown in figure 8 are the room temperature compression properties of quasi-isotropic AS4/PEEK tape (prepreg) laminate data from reference 10. The AS4/PEEK tow placed material compares closely with the AS4/PEEK tape material. The tape material was slightly stronger and stiffer (5% and 8% respectively).

When comparing the compression properties of the two tow placed composites, the AS4/PEEK was slightly stronger while the IM7/Radel had a significantly higher modulus. The tow placed AS4/PEEK was 8% stronger in un-notched compression strength and experienced a 35% greater ultimate strain than the tow placed IM7/Radel (see tables 3 and 4). The IM7/Radel compression modulus was 24% higher than that of the AS4/PEEK. Since compressive failure is strongly dependent on matrix properties, the higher compression strength and ultimate strain of the tow placed AS4/PEEK may be attributed to the higher modulus of PEEK compared to Radel (see table 2). The higher compressive modulus of the IM7/Radel may be explained by the higher modulus of the IM7 (43 Msi) fiber as compared to AS4 (36 Msi).

Both tow placed materials suffered a similar reduction in strength for notched compression (approximately 40%) and for hot/wet compression (approximately 15%). The AS4/PEEK specimens experienced 0.1% to 0.2% moisture gain during the 45 day soak of the H/W test while the IM7/Radel absorbed 0.4% to 0.5%. The IM7/Radel seemed more sensitive to the hot/wet notched testing as it retained only 46% of its room temperature un-notched compressive strength while the AS4/PEEK retained 55%. A similar trend was found

for the ultimate strains in H/W and notched compression (see tables 3 and 4). However, the moduli for both materials remained relatively unaffected by environmental testing (see figure 8).

The two tow placed quasi-isotropic materials were also tested in the 90° direction (see tables 3 and 4). One would expect the longitudinal (0°) and transverse (90°) properties to be comparable for a quasi-isotropic layup. While the 0° and 90° moduli were similar for both the AS4/PEEK and the IM7/Radel tow placed materials, both materials showed 6 to 7% higher strength in 90° compression than in 0° compression. A similar finding was reported for the AS4/PEEK tape laminate of reference 10 where the 0° and 90° direction strengths (both tension and compression) were significantly different. The higher compression strength in the 90° direction may be related to the fact that there are two 90° plies together at the center of the laminate. In effect there is one thick 90° ply at the midplane of the laminate and there is one less 90° ply interface than there are 0° ply interfaces.

Compression After Impact Properties

The room temperature and hot/wet compression after impact properties of the tow placed AS4/PEEK and IM7/Radel are shown in figure 9, along with the room temperature CAI data for AS4/PEEK tape from reference 11. The compression strengths (un-impacted) shown are repeated from figure 8 and the CAI data are from table 5 and reference 11. The numbers in parentheses at the top of the columns indicate the damage areas as determined from C-scans taken after the impact event. A target impact energy of 30 ft-lb (1500 in.-lb/in. of thickness) was used in the current investigation, as stated earlier. One AS4/PEEK specimen was inadvertently impacted at only about 15 ft-lb. This value is listed in table 5 but not included in figure 9.

As can be seen in figure 9, the CAI strength of the AS4/PEEK tow placed material compares closely with the AS4/PEEK tape material of reference 11, both being impacted with

an air gun at 1500 in.-lb/in. Although the actual impact energies varied somewhat (see table 5), and albeit the tow placed AS4/PEEK was somewhat stronger in un-notched compression, the tow placed IM7/Radel had a 26% greater room temperature CAI strength than the AS4/PEEK. The IM7/Radel retained 43% of its un-impacted strength while the AS4/PEEK retained only 32%. Even the one AS4/PEEK specimen inadvertently impacted at about 1/2 of the targeted impact energy was not stronger than the impacted IM7/Radel specimens. The higher strength of the IM7/Radel compared to AS4/PEEK is consistent with the smaller damage area (almost half) of the IM7/Radel. Some of this difference in strength between the two materials may be attributed to the different fibers. For example, reference 11 compared quasi-isotropic IM7/PEEK and AS4/PEEK tape laminates and found the IM7/PEEK to have approximately 10% greater CAI strength. While the higher strength of the IM7 fibers over AS4 may have contributed to the higher CAI strength of the IM7/Radel, the difference is large enough to suggest that the Radel matrix contributed more to the better CAI characteristics than the PEEK matrix. It is the properties of the matrix material which contribute most to impact and CAI behavior (ref. 11).

The hot/wet CAI data shown in figure 9 and tables 2-5 for the tow placed materials indicate that the IM7/Radel also retained more of its room temperature compressive strength in hot/wet CAI testing when compared to the AS4/PEEK. The IM7/Radel retained about 46% of its hot/wet un-impacted strength while the AS4/PEEK retained about 38% of its hot/wet un-impacted strength.

While the IM7/Radel material demonstrated smaller damage areas and higher CAI strengths, neither material demonstrated outstanding damage tolerance. A measure of merit for the CAI test is for the material to have a CAI strength of 40 ksi after a 1500 in.-lb/in. of thickness energy drop weight impact (ref. 12). While the materials of this study did not meet this measure of merit, the low CAI strengths may be partially attributed to the type of impact. The tow placed materials of this study were impacted with an air gun. When compared to drop weight impacting, it has been shown repeatedly that the higher velocity impact of an air gun firing an aluminum ball causes larger damage area and lower CAI strength. Reference 11 reported that quasi-isotropic AS4/PEEK tape laminates, impacted at 1500 in.-lb/in., had an

air gun CAI strength of about 31 ksi and a drop weight CAI strength of approximately 48 ksi, a 55% difference. It is important to note that the specimen geometry and boundary conditions during impact were also different. Reference 13 presents CAI data for two-phased toughened epoxy matrix materials subjected to both types of impact at 1500 in.-lb/in. The drop weight impact resulted in 33 to 55% higher CAI strengths and 50 to 61% smaller impact damage areas (ref. 13). These data suggest that direct comparisons between CAI data obtained from the two types of impact tests should be avoided. Although a drop weight type impact test was not performed for this work, in light of the results presented in reference 11, the air gun impacted CAI properties suggest that the ATP thermoplastic materials would meet the measure of merit.

CONCLUDING REMARKS

Two graphite/thermoplastic composite materials, AS4/PEEK towpreg and IM7/Radel 8320 slit tape, were used to make quasi-isotropic $[45/0/-45/9]_{ns}$ flat panels by automated tow placement. An attempt at auto-consolidation was made. Consolidation on-the-fly was found to be a complex but potentially viable process needing more research and development to become useable for full consolidation of thermoplastic composites. The tow placed composites of this study were partially consolidated in-situ and then fully consolidated with a more traditional vacuum bag/autoclave process.

Un-notched tension, notched tension, un-notched compression, notched compression, compression after impact and hot/wet tests were performed with the two tow placed composite materials. The properties of the tow placed quasi-isotropic AS4/PEEK laminates were compared with quasi-isotropic AS4/PEEK tape properties found in the literature. The tow placed AS4/PEEK material was somewhat stronger in tension but also somewhat weaker in compression than the AS4/PEEK tape laminates. The CAI properties of AS4/PEEK tape and tow placed materials were similar.

Comparing the two tow placed materials, the IM7/Radel demonstrated a significantly higher modulus in both tension and compression than the AS4/PEEK. The IM7/Radel also had a higher tensile strength. These better properties can be related to the higher strength and stiffness of the IM7 fiber as compared to the AS4. The AS4/PEEK was stronger in compression than the IM7/Radel. This higher compressive strength can be ascribed to the higher modulus of the PEEK resin as compared to Radel. The IM7/Radel outperformed the AS4/PEEK in all CAI tests.

While the CAI strengths did not indicate high levels of damage tolerance in these tests, this fact may be attributed in part to the type of impact test. These materials may have shown a significantly better CAI strength if tested with a drop weight impact instead of an air gun impact. The above findings suggest that these materials may be suitable for aircraft structure; however, the real potential of tow placed thermoplastics lies in the manufacturing process. If effective, low cost auto-consolidation can be successfully achieved, tow placed thermoplastics will compete more favorably with other materials under development.

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Table 1. Fiber Properties

Property	AS4	IM7
Tensile strength (ksi)	590	770
Tensile modulus (Msi)	36	43.6
Tensile strain (%)	1.65	1.81
Specific gravity	1.80	1.78
Cost 6K tow, 0-100 lbs(\$/lb)	28	62

Table 2. Resin Properties

Property	PEEK	Radel
Tensile yield strength (ksi)	13.6	11
Tensile modulus (Msi)	0.515	0.415
Tensile strain (%)	>40	100
Specific gravity	1.32	1.37
Tg (°F)	290	430
Processing temp. range (°F)	660-770	650-730

Table 3. AS4/PEEK Mechanical Properties, [45/0/-45/90]_{ns}

Property	n	Strength (ksi)	Modulus (Msi)	Ultimate strain (%)	Possion's ratio	Fiber vol. fract. (%)
Tension	3	97.5	6.60	1.51	0.31	53.9
Compression	6	87.3	6.10	1.67	0.32	57.2
90° compression	6	92.7	5.99	1.85	0.31	57.2
H/W compression	6	75.0	5.84	1.45	0.32	57.2
Notched tension	6	59.6*	6.66	0.76	--	57.2
Notched compression	6	55.1*	6.34	0.92	--	57.2
H/W notched compression	6	47.8*	6.14	0.81	--	57.2

* Strength based on gross cross-sectional area

Table 4. IM7/Radel Mechanical Properties, [45/0/-45/90]_{ns}

Property	n	Strength (ksi)	Modulus (Msi)	Ultimate strain (%)	Possion's ratio	Fiber vol. fract. (%)
Tension	3	130.0	8.68	1.48	0.31	63.6
Compression	6	79.9	7.59	1.08	0.33	64.0
90° compression	6	85.7	7.51	1.26	0.32	64.0
H/W compression	6	66.1	7.57	0.92	0.33	64.0
Notched tension	6	67.8*	8.33	0.81	--	64.0
Notched compression	6	46.6*	7.91	0.59	--	64.0
H/W notched compression	6	36.9*	7.75	0.49	--	64.0

* Strength based on gross cross-sectional area



Table 5. Compression After Impact Results, [45/0/-45/90]_{6s}

Specimen	Impact energy (in.-lb/in.)	Damage area (in.²)	Strength (ksi)
AS4/PEEK			
1*	770	1.91	33.3
2#	1410	5.19	28.2
3	1430	4.49	27.5
IM7/Radel			
1	1470	2.11	34.9
2	1520	2.82	34.3
3#	1530	2.60	30.5

Hot/wet test.

* Not included in average plotted in figures.

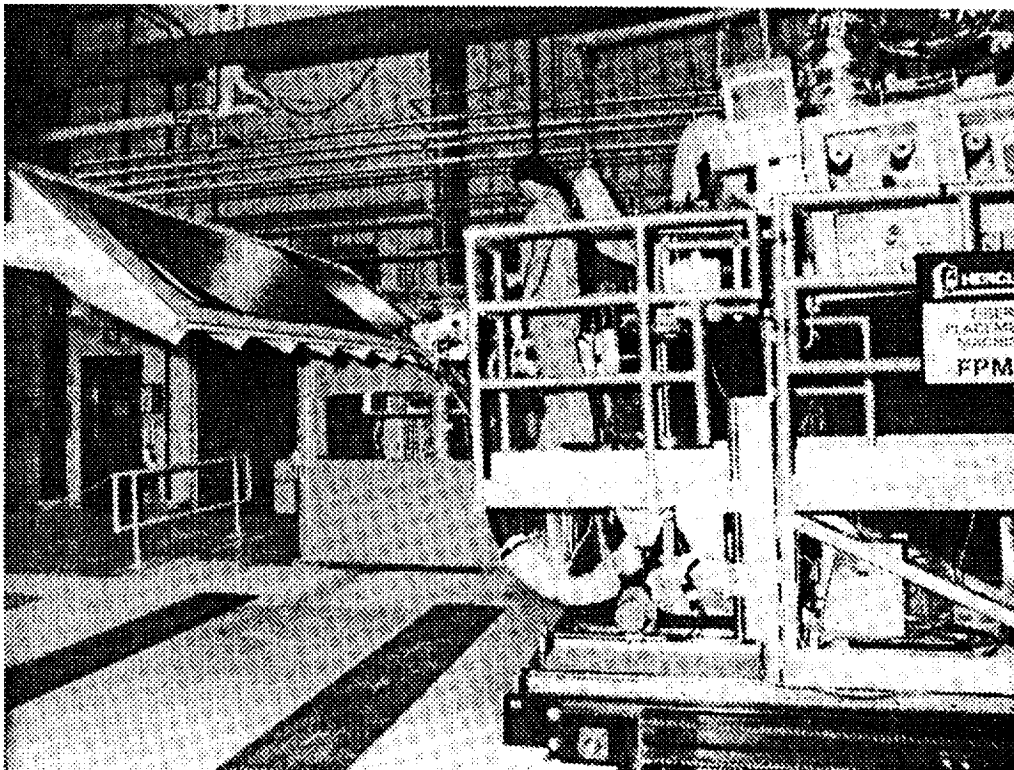
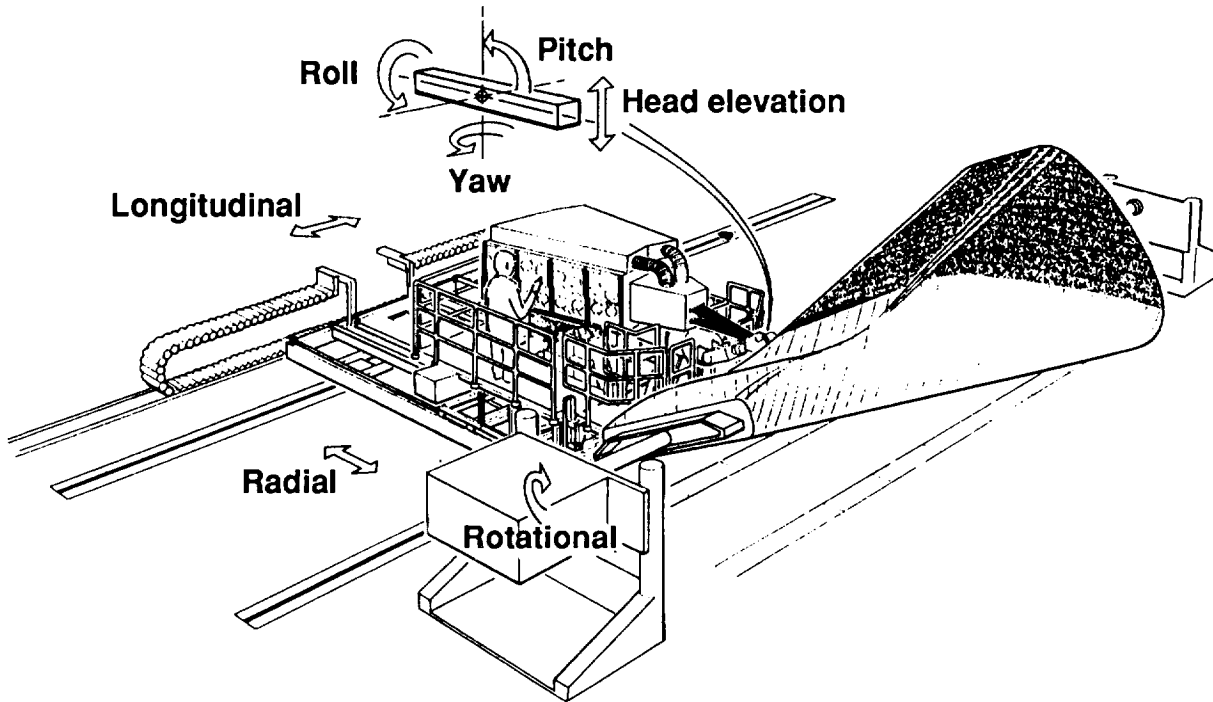


Figure 1. Automated tow placement machine, schematic and photograph (ref. 1).

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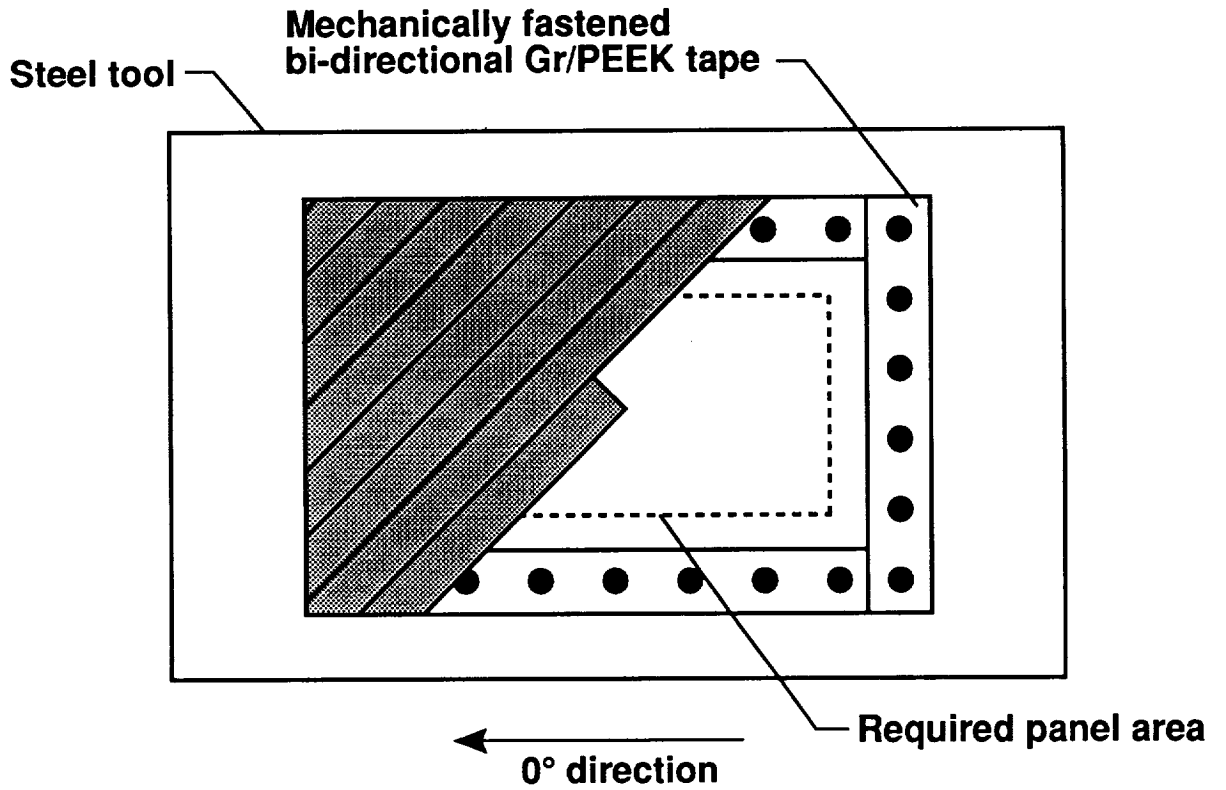


Figure 2. Schematic of first ply adhesion technique.

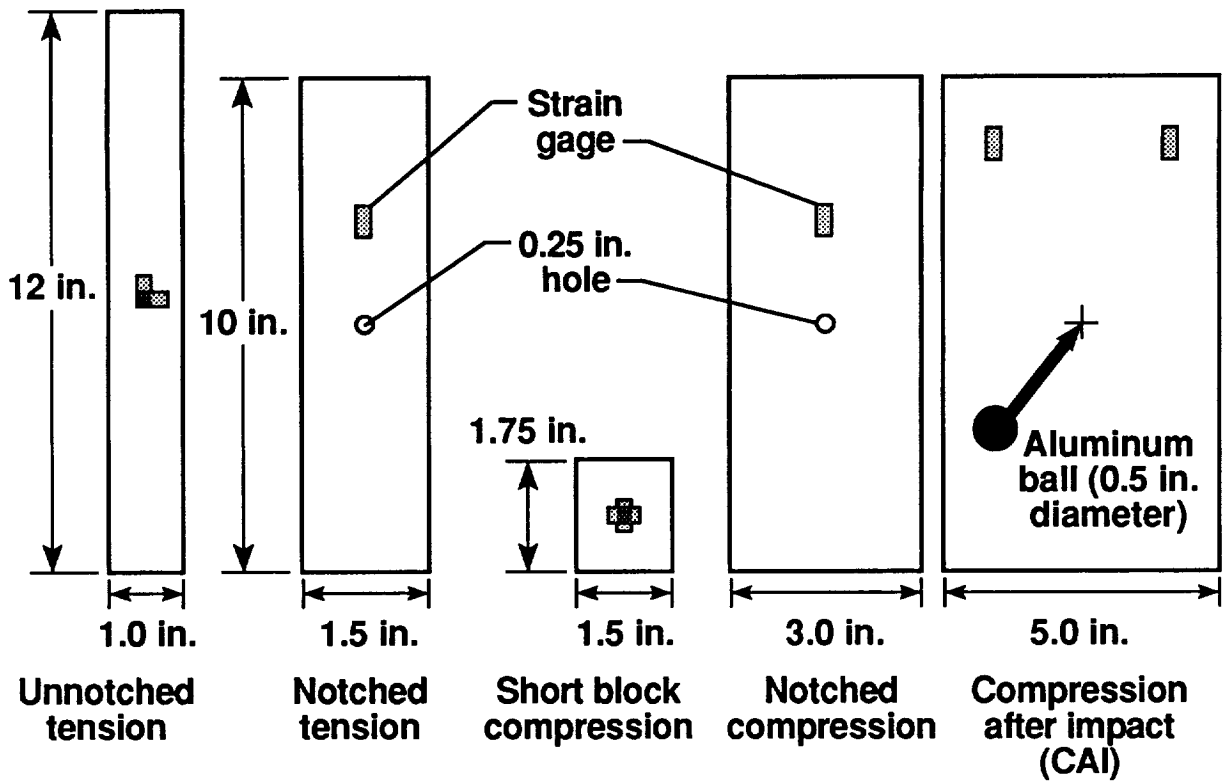


Figure 3. Test specimen configurations.

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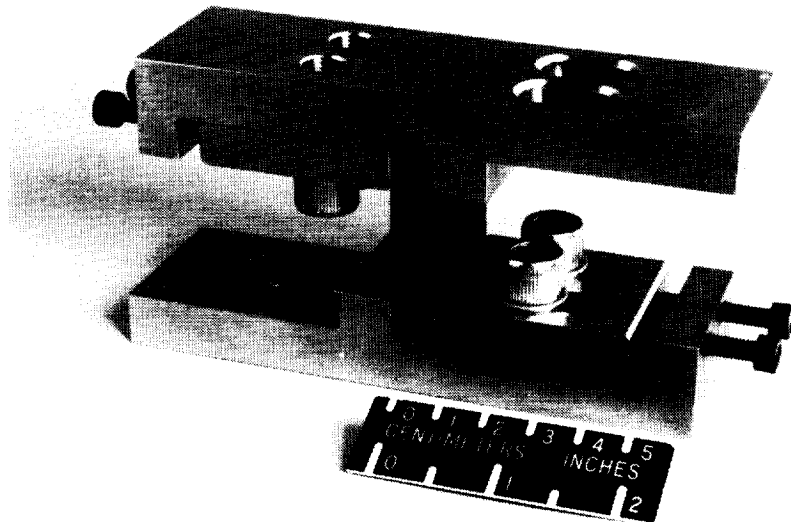


Figure 4. Short block compression test fixture.

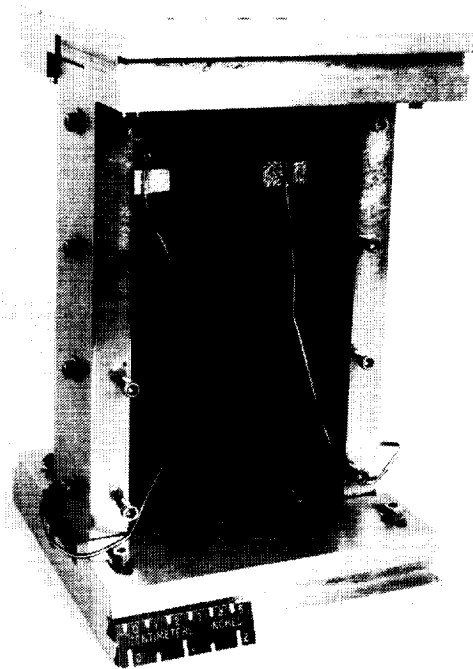


Figure 5. Compression after impact test fixture.

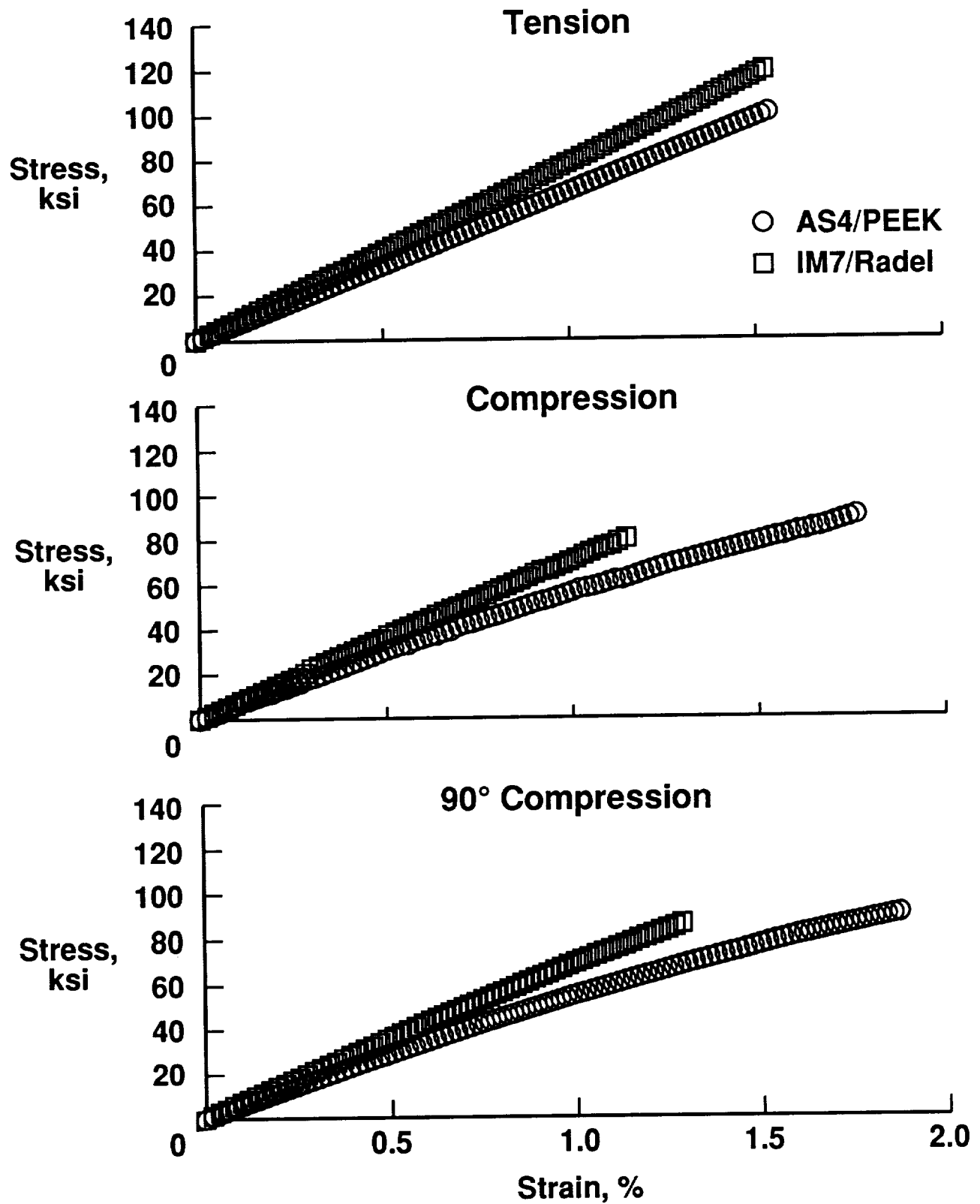


Figure 6. Typical stress-strain curves for tow placed quasi-isotropic graphite/thermoplastic composites.

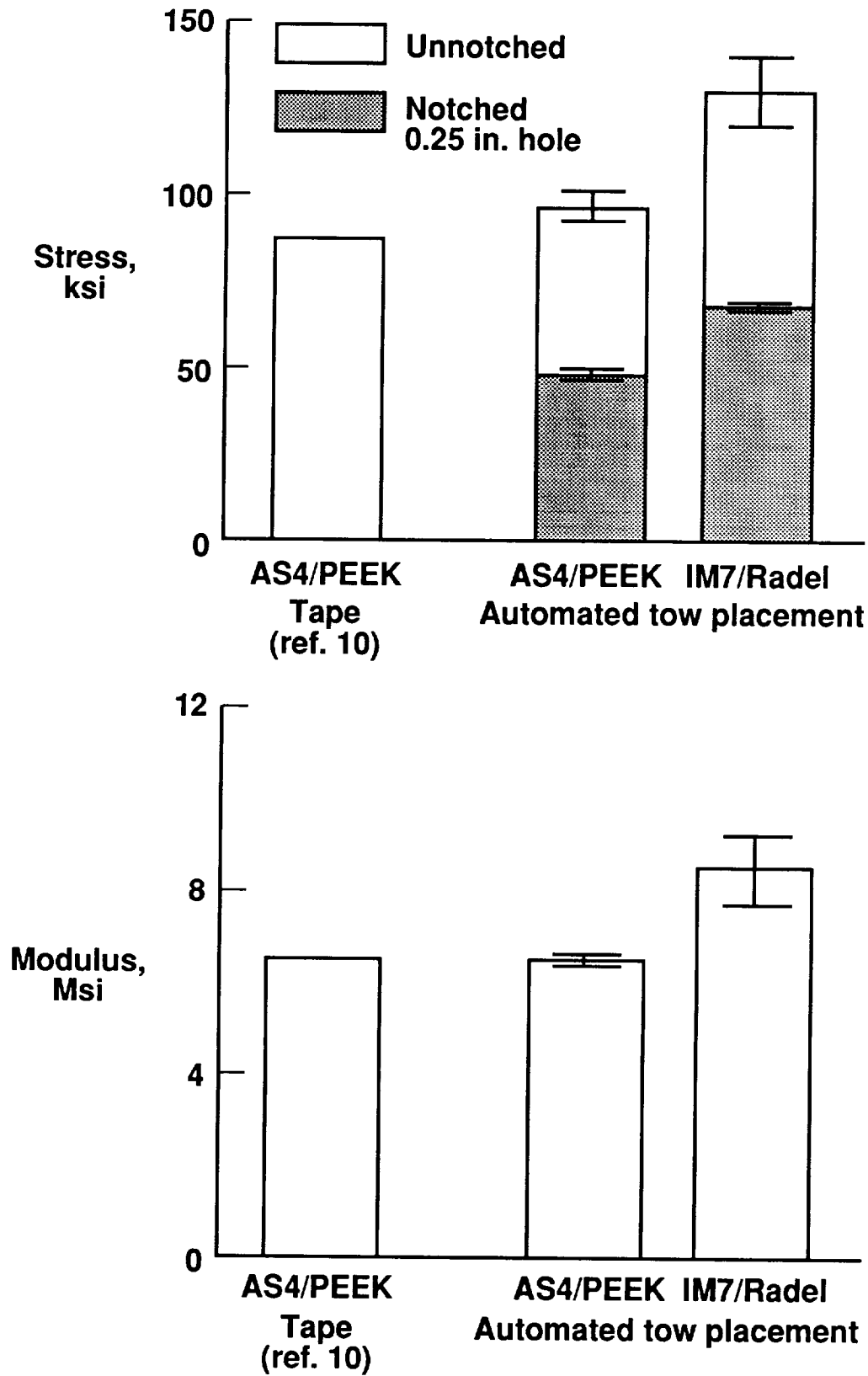


Figure 7. Notched and un-notched room temperature tension strengths and moduli of tow placed quasi-isotropic graphite/thermoplastic composites.

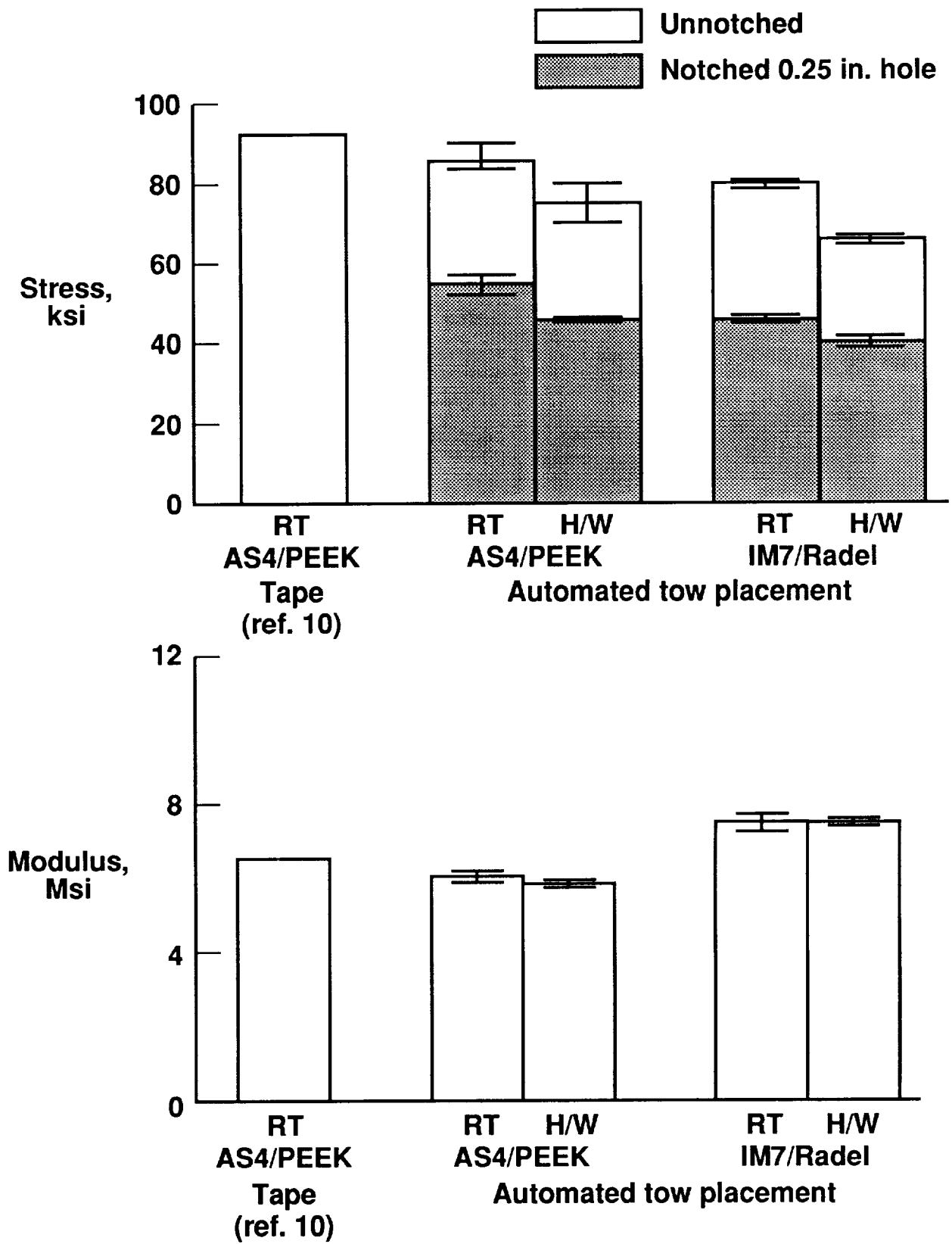


Figure 8. Notched and un-notched room temperature and hot/wet compression strengths and moduli of tow placed quasi-isotropic graphite/thermoplastic composites.

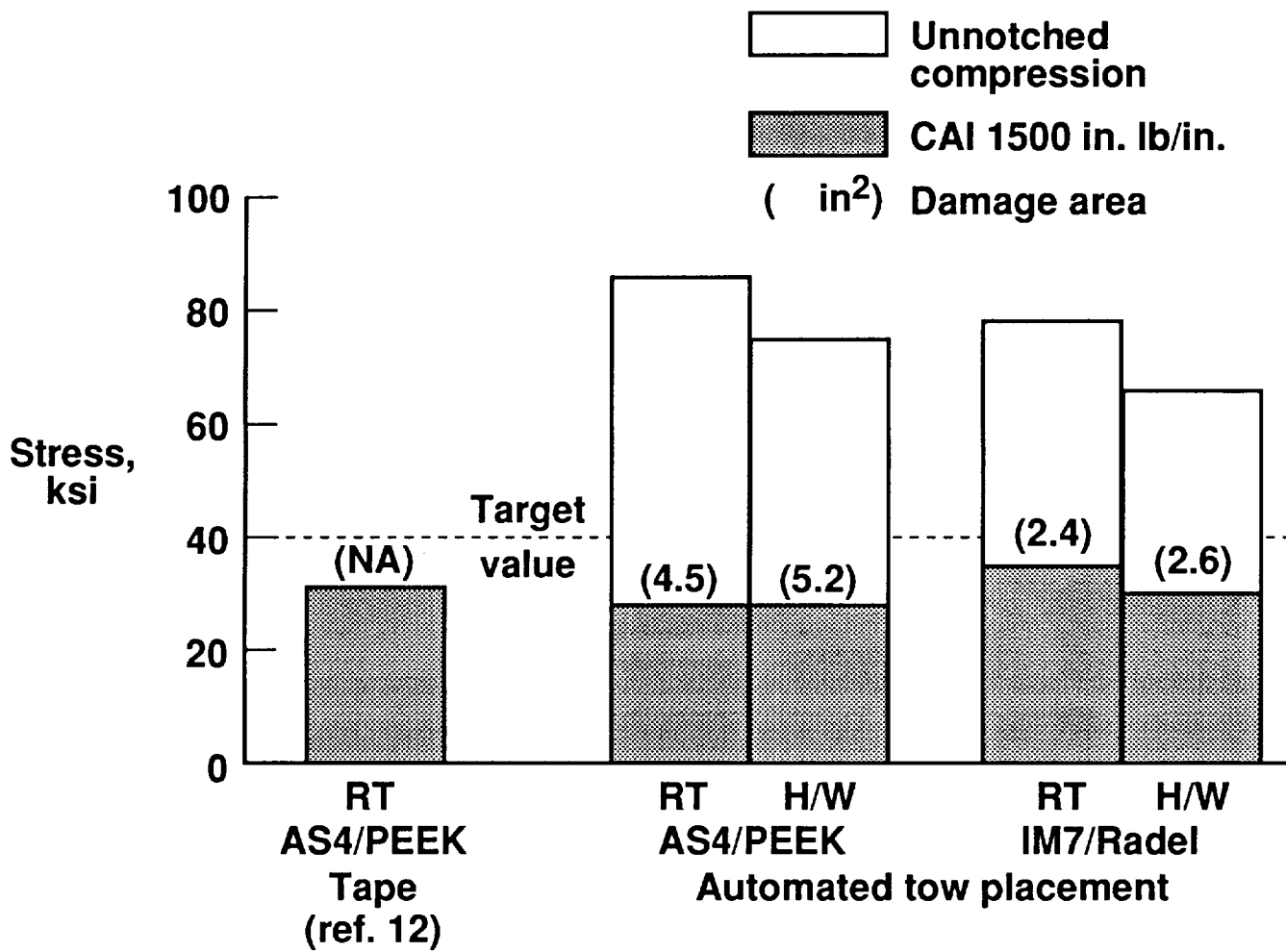


Figure 9. Compression and compression after impact strengths of tow placed quasi-isotropic graphite/thermoplastic composites.

