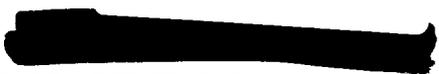


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THE EFFECTS OF AIRCRAFT FUEL AND FLUIDS ON THE STRENGTH PROPERTIES OF RESIN TRANSFER MOLDED (RTM) COMPOSITES*

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SUMMARY

The resin transfer molding (RTM) process offers important advantages for cost-effective composites manufacturing, and consequently has become the subject of intense research and development efforts. Several new matrix resins have been formulated specifically for RTM applications in aircraft and aerospace vehicles. For successful use on aircraft, composite materials must withstand exposure to the fluids in common use. The present study was conducted to obtain comparative screening data on several state-of-the-art RTM resins after environmental exposures were performed on RTM composite specimens. Four graphite/epoxy composites and one graphite/bismaleimide composite were tested; testing of two additional graphite epoxy composites is in progress. Zero-deg tension tests were conducted on specimens machined from eight-ply (+45-deg, -45-deg) laminates, and interlaminar shear tests were conducted on 32-ply 0-deg laminate specimens. In these tests, the various RTM resins demonstrated widely different strengths, with 3501-6 epoxy being the strongest. As expected, all of the matrix resins suffered severe strength degradation from exposure to methylene chloride (paint stripper). The 3501-6 epoxy composites exhibited about a 30% drop in tensile strength in hot, wet tests. The E905-L epoxy exhibited little loss of tensile strength (< 8%) after exposure to water. The CET-2 and 862 epoxies as well as the bismaleimide exhibited reduced strengths at elevated temperature after exposure to oils and fuel. In terms of the percentage strength reductions, all of the RTM matrix resins compared favorably with 3501-6 epoxy.

INTRODUCTION

The resin transfer molding (RTM) process offers important advantages for cost-effective composites manufacturing. Consequently, RTM is currently the subject of intense research and development efforts. One of the main advantages of RTM composite manufacturing is that the resin and reinforcement are not combined until a part is actually produced, eliminating the need for prior production of prepregged materials. Other advantages of RTM include the use of a wide variety of textile reinforcements such as woven, braided, and stitched preforms, and the ability to fabricate complex molded parts.

Several new matrix resins have been formulated specifically for RTM applications in aircraft and aerospace vehicles. For successful application in aircraft structures, composite materials must withstand exposure to fluids in use during operation and maintenance. Extensive studies have been conducted in the past with actual in-service exposure of graphite and boron/epoxy test coupons on operating aircraft (refs. 1, 2, and 3). The present study was conducted to obtain comparative screening data on several state-of-the-art RTM composites after environmental exposure.

*Work performed on Contract NAS1-18954 by Boeing Defense & Space Group.

Fluids in the operating environment can affect both the matrix resin and the fiber-matrix interface, thereby reducing composite properties. The fiber-matrix interface in RTM composites is formed during infiltration of resin into the preform and subsequent resin cure, and requires thorough wetting of the fibers by the resin. Of interest was the determination of what effect fluids would have not only on the resin matrix, but also on the resin-fiber interface in RTM composites, which could be different from the interface in composite structures produced from prepreg.

Composite tests that would give matrix-dominated failures were selected. Zero-deg tension tests were conducted on specimens machined from eight-ply (+45-deg, -45-deg) laminates, and interlaminar shear tests were conducted on 32-ply 0-deg laminate specimens. Tensile strength and modulus were measured; however, other properties could have been selected, such as tensile yield strength. There is no general agreement on which point should be selected on the stress-strain curve for property comparison and for relation to composite performance in service.

The panel preforms were produced from AS4 uniweave carbon fiber fabric. Four epoxy composites and one bismaleimide composite were tested; two additional graphite epoxy composites are in test.

EXPERIMENTAL

Zero-deg tension tests were conducted on (+45-deg, -45-deg) laminates per ASTM D3518 and D3039. Interlaminar short beam shear tests of 0-deg laminates were conducted per ASTM D2344. The tensile test specimens (Figure 1) were machined from 8-ply laminates with a nominal thickness of 1.14 mm (0.045 in); the interlaminar shear test specimens (Figure 2) were machined from 32-ply 0-deg laminates with a nominal thickness of 4.57 mm (0.180 in). Tensile test coupons were fabricated by bonding tapered E-glass/epoxy tabs (1.52 mm (0.060 in) thick) to rectangular blanks with epoxy adhesive, and then cutting specimens from these bonded blanks.

The laminates were layed up and resin transfer molded by the Douglas Aircraft Company from uniweave Hercules AS4 carbon fiber fabric. Five resins were evaluated (Table I), and panels from two additional resins are being resin transfer molded at Boeing for testing (Table I). Laminates were ultrasonically scanned to locate porosity or other defects. Some (+45-deg, -45-deg) laminates produced with E905-L epoxy had porosity in limited areas, possibly due to the location of injection and venting ports, and, where possible, specimens were cut outside of these porous areas. Fiber and void volume fractions were determined for selected panels (Table II).

The tensile and interlaminar shear test specimens were exposed to seven fluids (Table III). Some fluid exposures were conducted at an elevated temperature of 71°C (160°F), and tests were run at both ambient temperature and 82°C (180°F). The specimens exposed to JP-4 jet fuel were exposed at ambient temperature only, since special equipment and additional safety procedures would not be required. The specimens were exposed for 14 days (336 hours) to the fluids. The fluid exposures were accomplished by putting the specimens in cans filled with the fluid so that the specimens were fully immersed and, for elevated temperature exposures, the loosely capped cans were placed in an air-circulating oven at 71°C (160°F). Control specimens (unexposed) were placed in the same oven for 14 days.

The 14-day exposure period is somewhat arbitrary and was selected based on the time required for thin graphite/epoxy laminates to reach moisture saturation at 71°C (160°F). In the Boeing specification for secondary graphite epoxy structure, BMS 8-212 (ref. 4), a 14-day exposure period to moisture is used, while in the Boeing specification for toughened graphite/epoxy (ref. 5), a 6-day fluid exposure period is used. The exposure period is selected to produce a desired amount of fluid absorption in the composite test specimens. These time periods are not related to the lifetime exposures to which composite airplane

structures are subjected. The testing performed in this effort was intended to provide a relative indication of composite performance.

The ends of some of the specimens were wrapped with aluminum foil and aluminum foil tape to reduce solvent absorption by the tabs and tab adhesive; only the specimen gauge area was exposed to the solvent. The short-beam shear specimens were held within small folded wire screen sections to keep specimens from the same material grouped together.

Weight gain was measured to determine the percent of fluid absorbed by some of the short beam shear specimens (Table IV). The percent of weight gain was usually small, except for water at 71°C (160°F) and methylene chloride. Additional weight gain measurements will be obtained to confirm this data.

Five specimens were mechanically tested for each material, fluid, and condition. Both shear strength and shear modulus were measured for the tensile specimens. The modulus was measured for only two of the five specimens in each group. A biaxial extensometer (Figure 3) was used to measure displacement in both the axial and transverse directions to determine the shear modulus. All tensile specimens were tested to failure, which usually took 30 to 40 minutes per specimen due to the high elongation of the specimens. A crosshead speed of 1.27 mm (0.05 in.) per minute was used. One of the test machines is shown in Figure 4, and the environmental chamber used to heat the specimens is shown in Figure 5.

RESULTS AND DISCUSSION

The mechanical test results for all groups of specimens are summarized in bar charts for the tensile test specimens (Figures 6 through 10) and for the interlaminar shear specimens (Figures 11 through 15). In general, test results indicated that all of the composites retained adequate strength after exposure to JP-4 jet fuel, commercial hydraulic fluid, turbine oil, methyl ethyl ketone, deicing fluid, and water. Methylene chloride exposure usually caused significant deterioration of mechanical properties.

Tensile coupons immersed in methylene chloride usually retained only 50% to 60% of the room-temperature control specimens' shear strength with the exception of AS4/3501-6 epoxy, which retained 80% of its strength. Decreases in interlaminar shear strength similar to tensile strength degradation did not occur for all composites tested, however. Since methylene chloride is readily absorbed by polymeric materials, it should not be used around composite aircraft structures.

Other than methylene chloride, water at 82°C (180°F) caused the next largest drop in properties, usually a 10% to 25% decrease in shear strength. The AS4/CET-2 epoxy, however, showed no drop in shear strength under this hot/wet exposure, and the AS4/3501-6 showed its greatest drop (24%) in shear strength. Interlaminar shear strength typically showed a 10% reduction at 82°C (180°F) after 14 days exposure in 71°C (160°F) water.

The AS4/CET-2 material exhibited a 16% drop in shear strength after exposure to hydraulic fluid and testing at 82°C (180°F). A similar decline was not observed in the interlaminar shear strength, however.

It could not be determined why the AS4/5292 bismaleimide control specimens had a low room temperature shear strength. The ambient interlaminar shear strength did not show a similar trend. Exposure to JP4 jet fuel also caused an appreciable (19%) decrease in shear strength of AS4/5292.

JP4 jet fuel did not cause significant property deterioration in the RTM composites under these exposure conditions, however, more significant deterioration can occur with longer exposures (ref. 6). Work at Boeing on military airplane programs has indicated that the water that collects in fuel systems is more detrimental to composite properties than the fuel itself.

CONCLUSIONS

In these tests, the various RTM resins demonstrated widely different strengths, with 3501-6 epoxy being the strongest. As expected, all of the matrix resins suffered severe strength degradation from exposure to methylene chloride (paint stripper). The 3501-6 epoxy composites exhibited about a 30% drop in tensile strength in hot, wet tests. The E905-L epoxy exhibited little loss of tensile strength (< 8%) after exposure to water. The CET-2 and 862 epoxies as well as the bismaleimide exhibited reduced strengths at elevated temperature after exposure to oils and fuel.

In terms of the percentage of strength reductions, all of the RTM matrix resins compared favorably with 3501-6 epoxy. By inference, an adequate fiber-matrix interface is apparently forming in these RTM composites, indicating that the fibers are sufficiently wet-out with resin in the RTM process.

ACKNOWLEDGEMENTS

This work was performed under contract from the NASA Langley Research Center (Contract NAS1-18954) and their support is gratefully acknowledged. Resin transfer molded laminates were supplied by the Douglas Aircraft Company; Mr. Raymond Palmer of Douglas Aircraft and Mr. William Avery of the Boeing Defense & Space Group provided recommendations on fluid exposure testing. Boeing technicians, Mr. Oscar Davis and Mr. Allen Kelly, conducted the mechanical tests. Ms. Erica Smith, a summer intern at Boeing, measured most of the specimens and performed the initial mechanical property calculations.

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5. Boeing Material Specification 8-276: Advanced Composites - 350°F (177°C) Cure Toughened-Epoxy Preimpregnated Carbon Fiber Tapes and Fabrics, The Boeing Company, Revised Nov. 1991.
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Table I. RTM Matrix Resins Under Investigation

Carbon fiber fabric used with all resins: 3K AS4 uniweave fabric at 145 g/m².

Testing has been completed on:

Manufacturer	Matrix resin	Identification code
Dow Chemical Company	Tactix* CET-2 Epoxy	D
Shell Chemical Company	Epon** DPL 862 Epoxy/Curing Agent W	S
Hercules	3501-6 Epoxy	H
BP Chemicals	E905-L Epoxy	B
Ciba Geigy Corp.	Matrimid*** 5292 Bismaleimide	C

Testing is in progress on:

Manufacturer	Matrix resin	Identification code
Dow Chemical Company	Tactix* CET-3 Epoxy	T
Shell Chemical Company	RSL 1895 Epoxy/Curing Agent W	R

*Trademark of the Dow Chemical Company

**Trademark of the Shell Chemical Company

***Trademark of Ciba Geigy Corp.

Table II. Selected RTM Laminate Physical Properties*

Laminate	Density (g/cm ³)	Fiber vol. fraction (%)	Void content (%)
0-deg, 32 plies, AS4/E905-L epoxy	1.59	61	< 1
0-deg, 32 plies, AS4/5292 bismaleimide	1.58	57	< 1
0-deg, 32 plies, AS4/CET-2 epoxy	1.77	55	< 1
±45-deg, 8 plies, AS4/E905-L epoxy	1.49	52	3
±45-deg, 8 plies, AS4/CET-2 epoxy	1.75	46	1

*Calculations based on an AS4 carbon fiber density of 1.80 g/cm³ and resin densities of 1.24 g/cm³ for E905-L epoxy, 1.23 g/cm³ for 5292 bismaleimide, and 1.75 g/cm³ for CET-2 epoxy.

Table III. RTM Laminate Exposure Fluids and Temperatures

Code Number	Fluid	Exposure temp.	Test temp.
1	JP-4 jet fuel	Ambient	Ambient
2	Chevron HyJet IVA Hydraulic Fluid	71°C (160°F)	Ambient
3	MIL-L-7808 Turbine Oil	71°C (160°F)	Ambient
4	Methyl ethyl ketone	Ambient	Ambient
5	Methylene chloride	Ambient	Ambient
6	Deicing fluid	Ambient	Ambient
7	Water	71°C (160°F)	Ambient
8	Control (no fluid exposure)	71°C (160°F)	Ambient
9	JP-4 jet fuel	Ambient	82°C (180°F)
10	Chevron HyJet IVA Hydraulic Fluid	71°C (160°F)	82°C (180°F)
11	MIL-L-7808 Turbine Oil	71°C (160°F)	82°C (180°F)
12	Water	71°C (160°F)	82°C (180°F)
13	Control (no fluid exposure)	71°C (160°F)	82°C (180°F)

Table IV. Percent Weight Gain of 32-Ply Unidirectional Laminate Coupons

Laminate	Exposure fluid and temperature (table III)												
	1	2	3	4	5	6	7	8	9	10	11	12	13
AS4/5292	0.06	0	0	0	0.08	0.08	1.15	N/A	0.05	0	0	1.11	N/A
AS4/CET-2	0	1.76	0.02	1.42	**	0.06	0.16	N/A	0.03	1.67	0.05	0.12	N/A
AS4/3501-6	0.07	**	**	0	**	0.07	**	N/A	0.06	0	0.05	**	N/A
AS4/862/W	0.04	0.06	0	**	4.42	0.18	**	N/A	0.12	0.07	0.01	**	N/A

*Values for the AS4/E905-L epoxy laminates were high (6% to 8%) and were probably in error.

**Not measured.

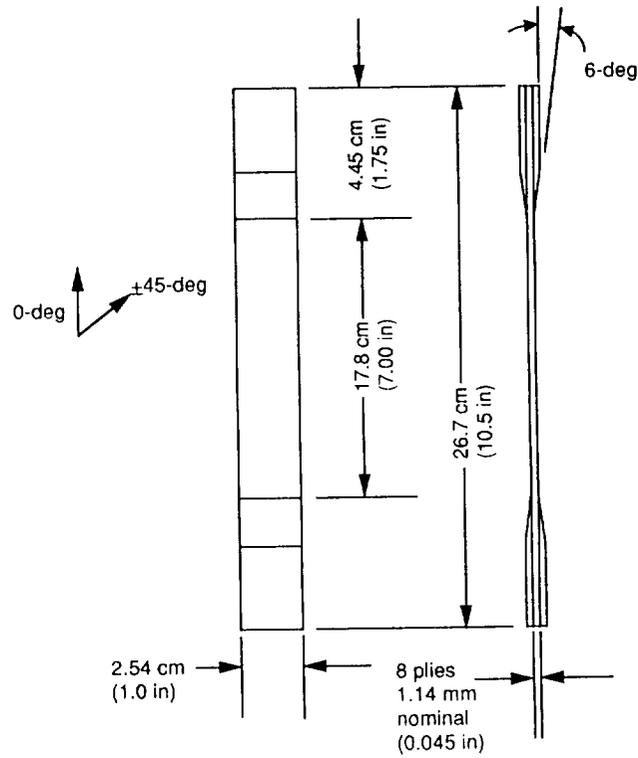


Figure 1. A ± 45 -deg tensile test specimen with bonded tabs, after ASTM D3518 and D3039.

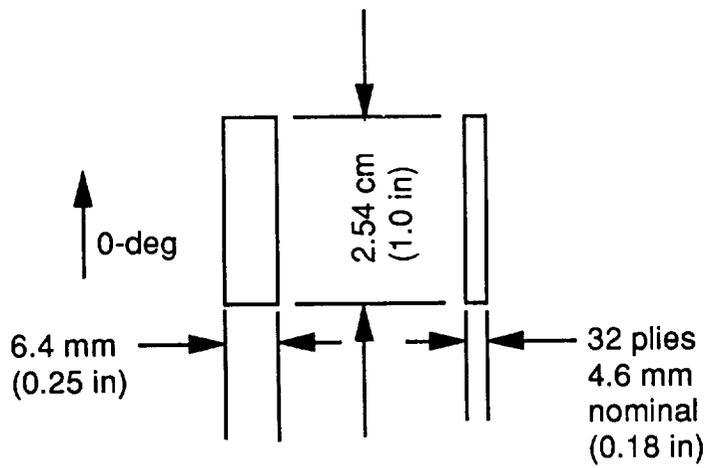


Figure 2. A 0-deg interlaminar shear test specimen, after ASTM D2344.

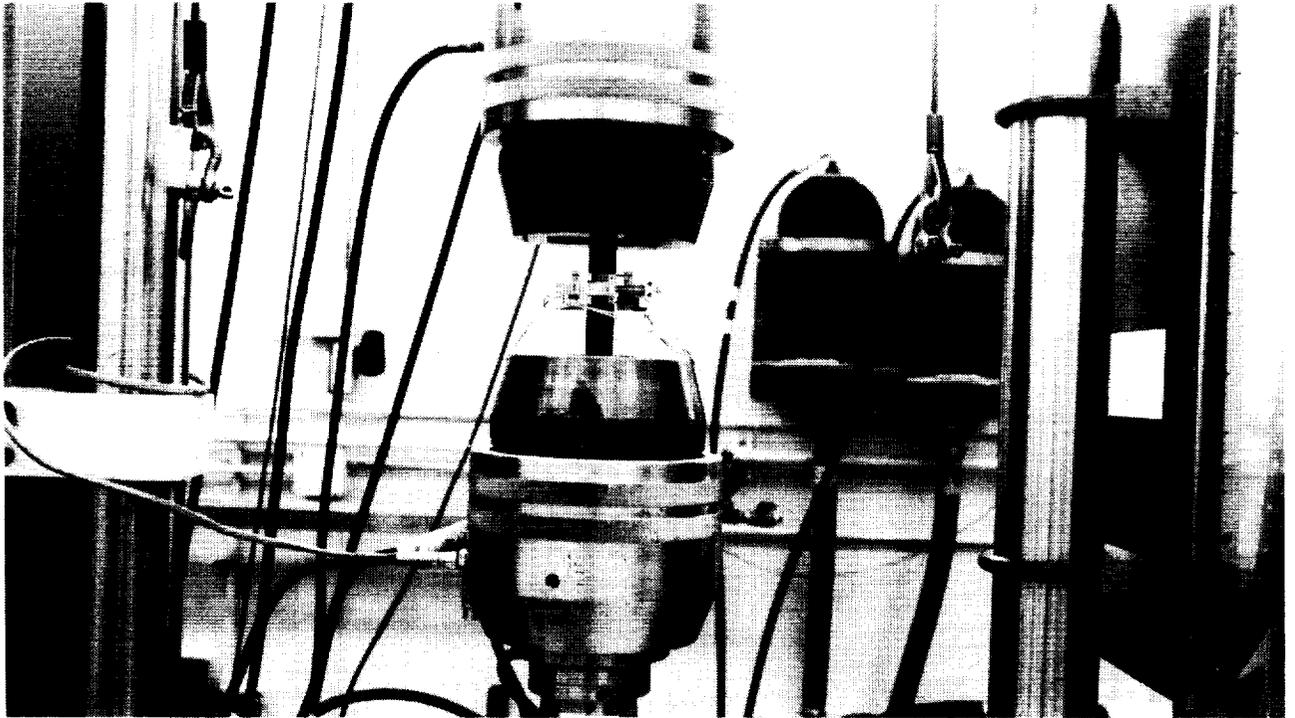


Figure 3. Biaxial extensometer on a tensile test specimen.



Figure 4. Mechanical test machine.

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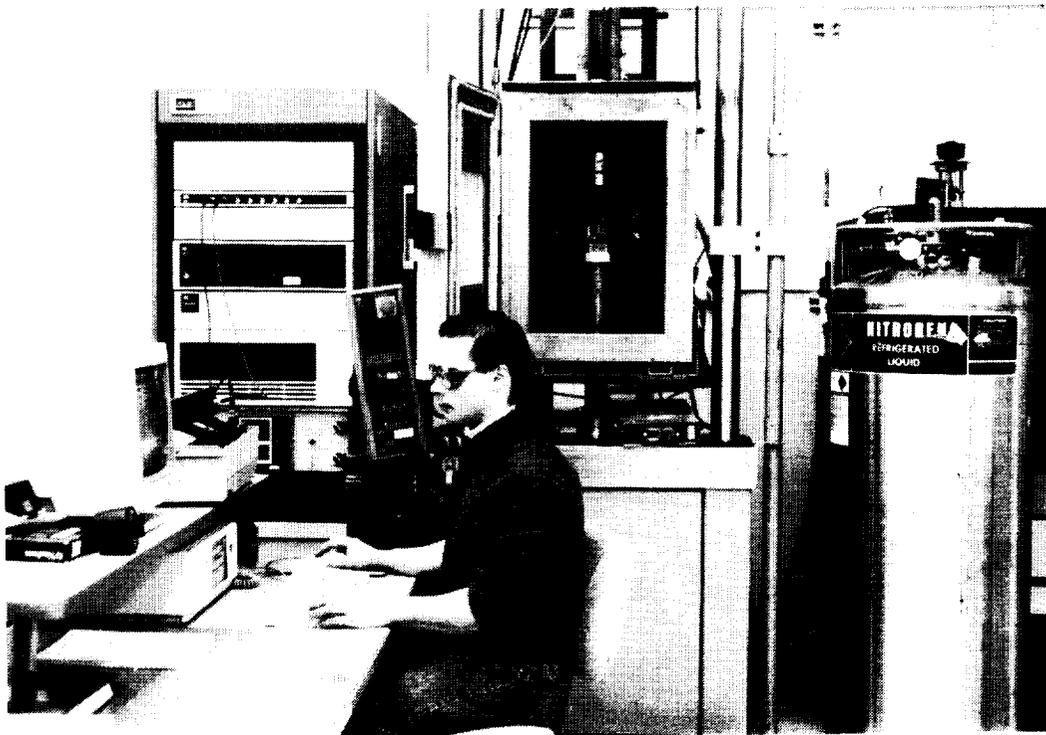


Figure 5. Mechanical test machine with environmental chamber.

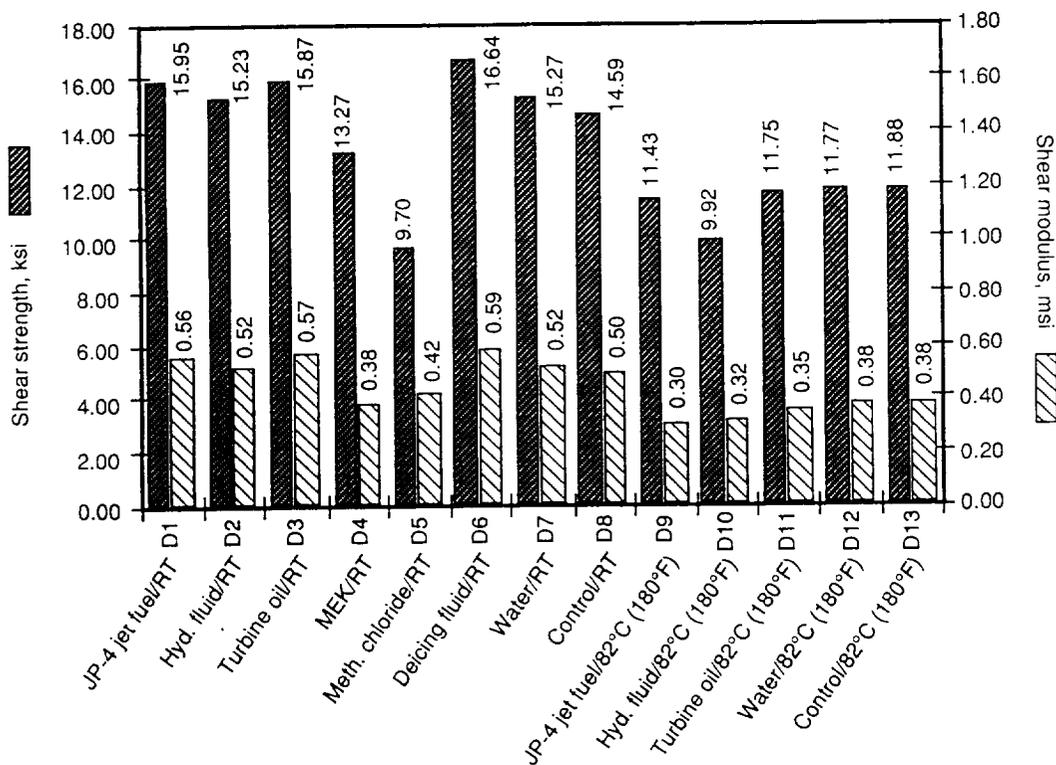


Figure 6. Average shear strength and shear modulus from ± 45 -deg tensile coupons of AS4/Dow Tactix CET-2 epoxy after fluid exposures.

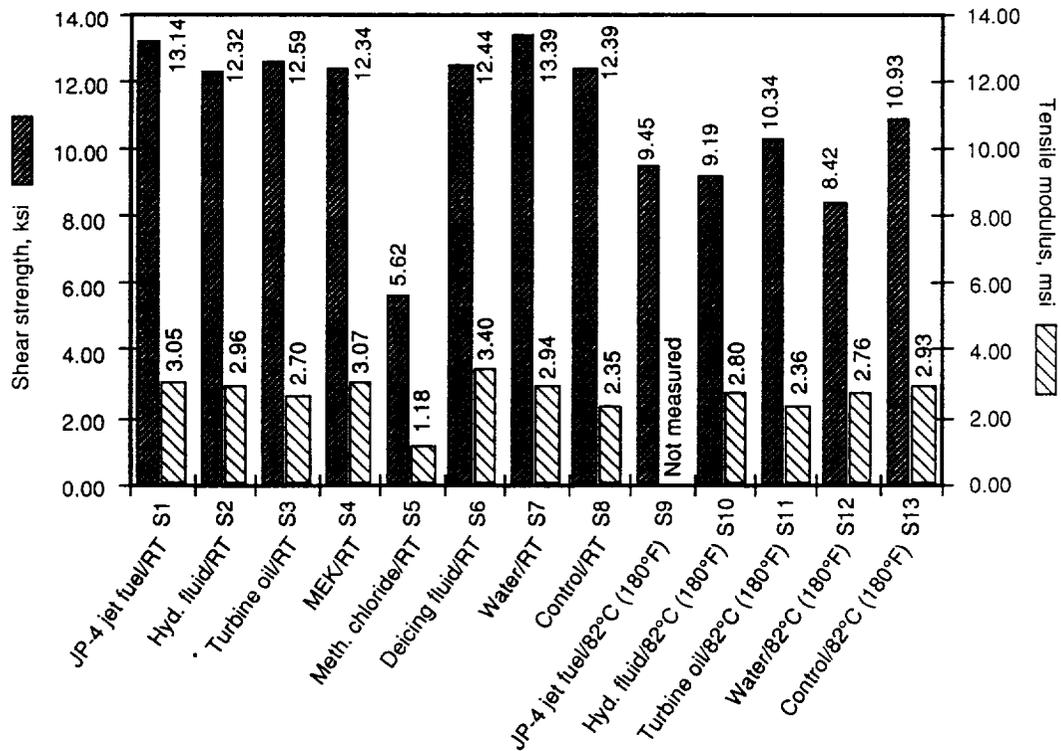


Figure 7. Average shear strength and shear modulus from ± 45 -deg tensile coupons of AS4/Shell Epon DPL 862/W epoxy after fluid exposures.

Note: Tensile modulus, not shear modulus (G_{12}), was measured.

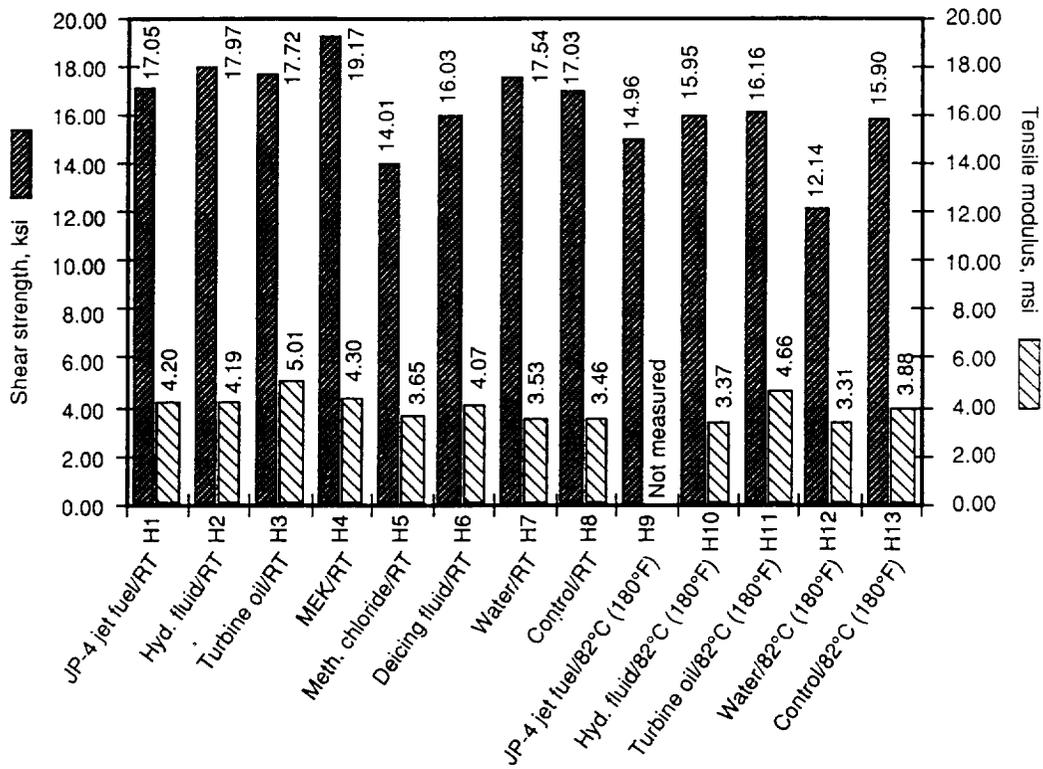


Figure 8. Average shear strength and shear modulus from ± 45 -deg tensile coupons of AS4/Hercules 3501-6 epoxy after fluid exposures.

Note: Tensile modulus, not shear modulus (G_{12}), was measured.

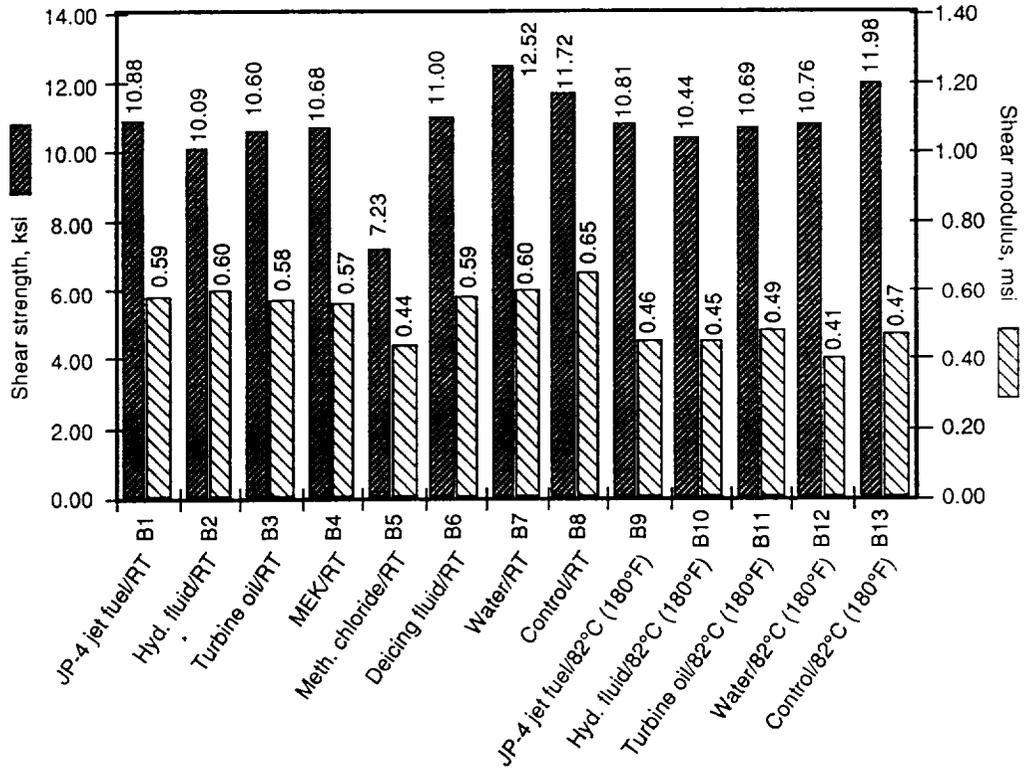


Figure 9. Average shear strength and shear modulus from ± 45 -deg tensile coupons of AS4/BP Chemicals E905-L epoxy after fluid exposures.

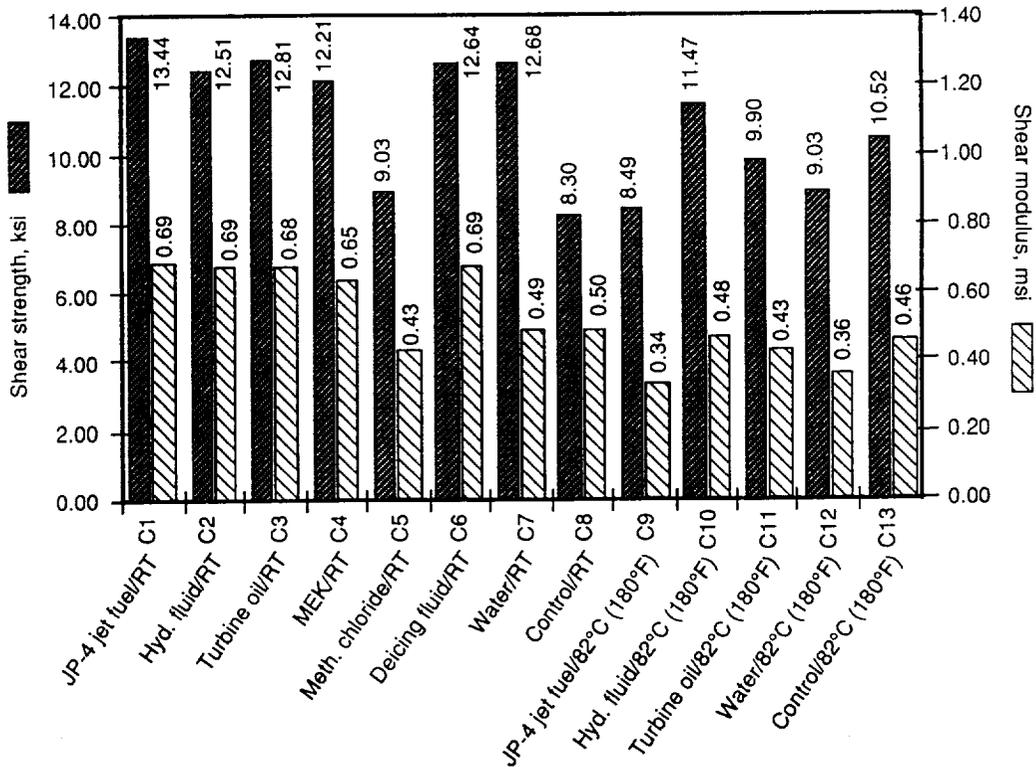


Figure 10. Average shear strength and shear modulus from ± 45 -deg tensile coupons of AS4/Ciba Geigy Matrimid 5292 bismaleimide after fluid exposures.

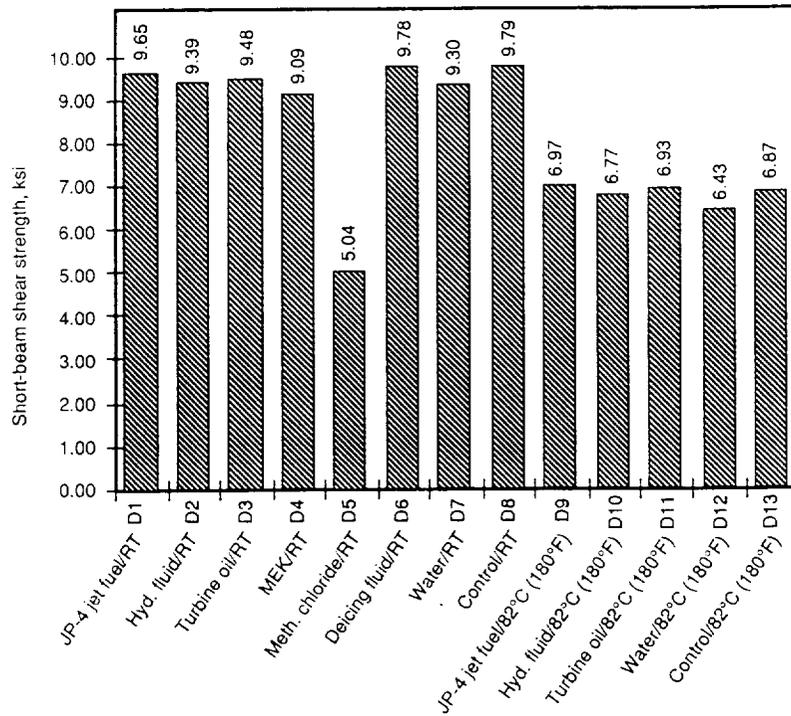


Figure 11 Average interlaminar shear strength of AS4/Dow Tactix CET-2 epoxy after fluid exposures.

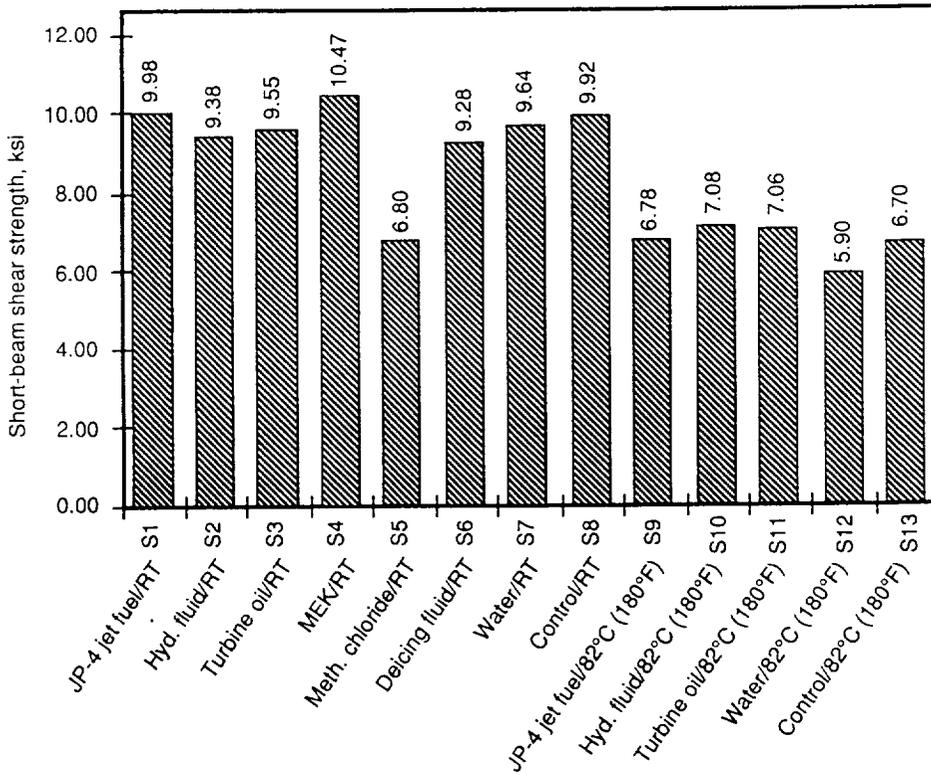


Figure 12. Average interlaminar shear strength of AS4/Shell Epon DPL 862/W epoxy after fluid exposures.

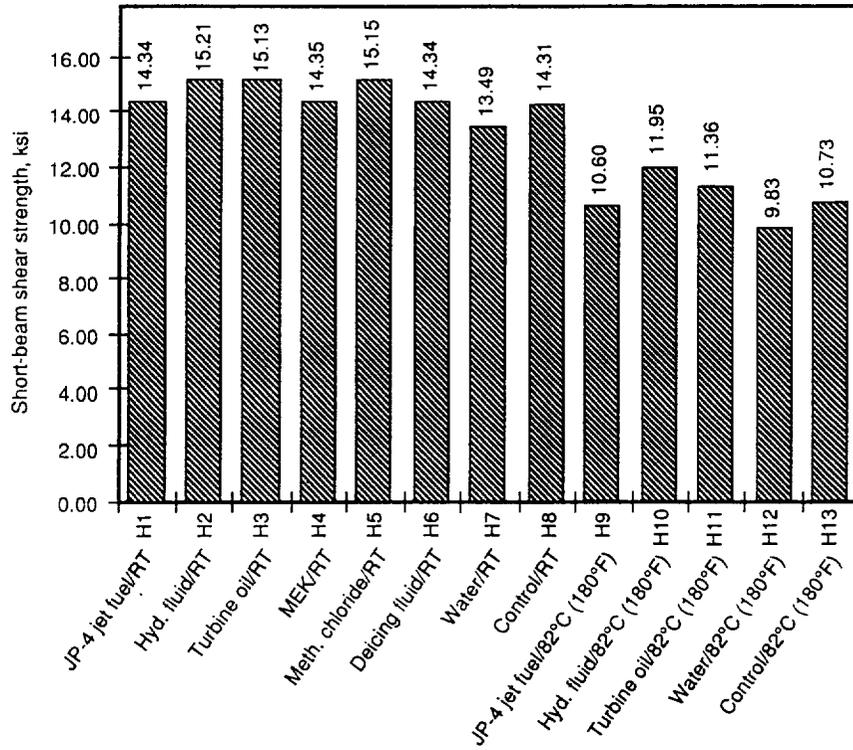


Figure 13. Average interlaminar shear strength of AS4/Hercules 3501-6 epoxy after fluid exposures.

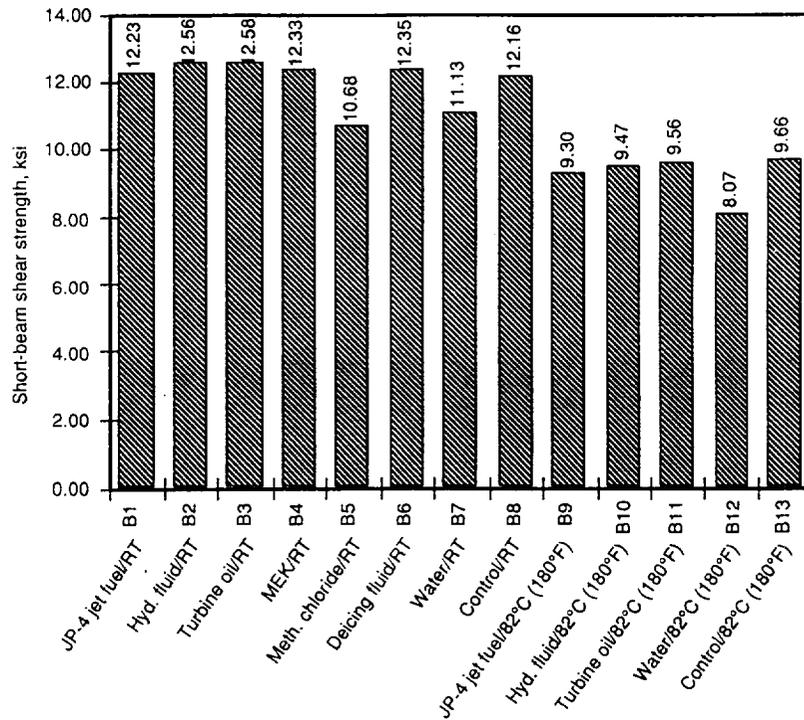


Figure 14. Average interlaminar shear strength of AS4/BP Chemicals E905-L epoxy after fluid exposures.

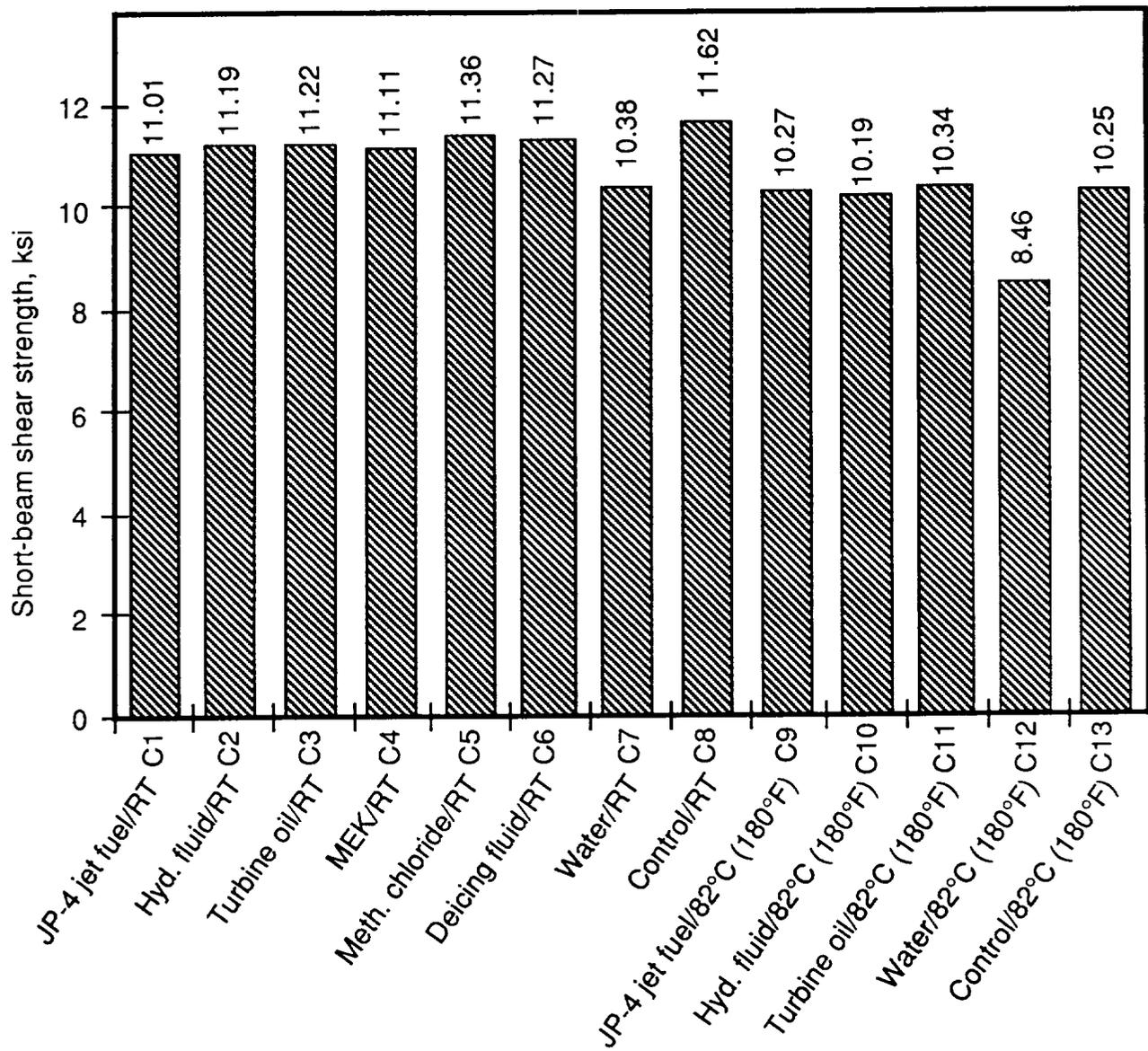


Figure 15. Average interlaminar shear strength of AS4/Ciba Geigy Matrimid 5292 bismaleimide after fluid exposures.

