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INTRINSICALLY SURVIVABLE STRUCTURAL COMPOSITE MATERIALS

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This program was initiated to develop baseline material properties to compare against properties of newly developed nanocomposites. An evaluation of the effects of strain rate and temperature on the in-plane shear properties of AS4/3501-6 and AS4/APC-2 was conducted. These tests indicate that the strain-to-failure and toughness of the AS4/3501-6 is highly strain rate dependent but the AS4/APC-2 is not. Budget cuts resulted in early termination of the program.

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FOREWORD

This report was prepared by the University of Dayton Research Institute under Air Force Contract No. F33615-95-D-5029, Delivery Order No. 0005. The work was administered under the direction of the Nonmetallic Materials Division, Materials and Manufacturing Directorate, Air Force Research Laboratory, Air Force Materiel Command, with Dr. James R. McCoy (AFRL/MLBC) as Project Engineer.

This report was submitted in January 1999 and covers work conducted from 15 September 1997 through 14 September 1998.

EXECUTIVE SUMMARY

The development of efficient and economical unoccupied combat aerial vehicles (UCAVs) requires revolutionary design and engineering of materials and structures that are capable of surviving static and dynamic loads at strain rates which vary over several orders of magnitude. These vehicles will operate in environments of greater hostility and threat than manned vehicles, and must be designed to accomplish their mission and return home after being hit by enemy fire. In addition they must possess the inherent requirements of today's conventional aircraft: high performance, lightweight, and low cost. These requirements may be met with efficiently designed polymer composite structures fabricated from intrinsically-survivable materials systems.

The original scope of this delivery order was to generate baseline data on conventional composite laminates which could then be used to compare against the performance of new nanocomposite toughened resin systems. The baseline data to be generated on a conventional epoxy system, AS4/3501-6, and a thermoplastic, AS4/APC-2, were to include in-plane shear properties at strain rates of 0.01-10.0/min and impact tests ranging from 1-1000 m/sec. Unfortunately, due to budget cuts only low speed in-plane shear properties were evaluated. These tests indicate that the strain-to-failure and toughness of the AS4/3501-6 is highly strain rate-dependent, but the AS4/APC-2 is not. In addition to characterization of the conventional composite materials, numerous characterization studies were conducted on nanocomposite samples in support of an Air Force visiting scientist.

1. TEMPERATURE AND STRAIN RATE EFFECTS ON COMPOSITE IN-PLANE SHEAR PROPERTIES

A test plan for investigating strain rate effects on the shear properties of both AS4/3501-6 and AS4/APC-2 composites was partially completed. This plan was designed to determine whether or not the principles of time-temperature superposition could be applied towards the characterization of a material's response toward ballistic impact. The fundamental difference of this application of time-temperature superposition is that the materials fracture energy is being investigated, not its viscoelastic modulus. Work conducted by Bitner *[1]* showed that a master curve using principles of time-temperature superposition could be developed for describing the fracture energy of neat resin adhesives.

The primary purpose for conducting this investigation was to develop a test method for screening the ballistic performance of small quantities of novel nanocomposite matrix resins. Experimental methods of conducting very-high strain rate tensile tests are complex and expensive, while ballistic tests require large amounts of material and a very large test matrix due to the near infinite variety in impact variables (composite lay-up, boundary conditions, projectile shape, projectile speed, temperature, etc.). In an effort to bound the range for fracture energy of conventional aerospace composite materials, a second-generation epoxy, 3501-6, and a high-performance thermoplastic, APC-2, were selected for investigation. Fortunately, both of these materials were readily available on AS4 carbon fiber. The mode I fracture energy for AS4/3501-6 and AS4/APC-2 is 86 and 1330 J/m^2, respectively, while the mode II fracture energy is 683 and 2625 J/m^2, respectively.

1.1 Size Effect on In-Plane Shear Properties

Delamination of composite laminates during ballistic impact is caused primarily by shear deformation *[2,3]* arising from component deflection which is inducted by the projectile force. We might expect, therefore, that measuring shear properties of composite laminates may provide a means of ranking delamination resistance of various composite materials. A standard test method, ASTM D3518-76, was chosen as a suitable test method for evaluating the composite shear properties. This method prescribes that shear properties be evaluated by applying tensile stress to specimens with a $[45/-45]_{2S}$ construction. Typically, such a test would be conducted on a specimen 2.54 cm wide with a 15.2-cm gage length; however, as a means of maximizing strain rate during the test, a smaller specimen is desirable. A set of tests were therefore conducted to determine the size effect on in-plane shear properties. Table 1 shows the test results for AS4/3501-6 tested with specimen geometries of 2.54 cm x 15.2 cm and 0.64 cm x 3.0 cm. During a strain rate of 1/minute, the larger geometry yielded a strength of 181 (5.5) MPa and modulus of 17.9 (1.4) GPa; while the smaller geometry yielded a strength of 178 (5.5) MPa and modulus of 20.0 (2.1) GPa. These differences do not appear to be significant given the standard deviation of the data. The more significant size effect was the ultimate strain-to-failure. The larger geometry yielded a strain to failure of 9.1 percent, while the smaller geometry failed at 4.0 percent strain. The severe necking which occurs during this test was interrupted by the end tabs on the sample with the small gage section, so this precipitated the earlier failure. For the case of AS4/APC-2 the results were similar. The ultimate strain-to-failure for the larger geometry was 24.8 percent, while the smaller geometry failed at 15.2 percent. These results indicate the specimen failure mode is geometry dependent and, therefore, it is important to only compare properties generated on samples of similar geometry. Further testing was restricted to samples of the smaller geometry to facilitate higher strain rates, conservation of material, and compatibility with split-Hopkinson bar results.

TABLE 1
In-Plane Shear Properties of AS4/3501-6

SAMPLE	S. RATE	TEMP, C	NUMBER	ULT STRESS, MPa	YOUNG'S MODULUS, GPa	%STRAIN @ MAX LOAD	NORMALIZED WORK, MPa	ULTIMATE STRAIN
AEM	0.01/MIN	AMBIENT	5	175 (7.6)	17.9 (2.1)	8.1 (0.1)		8.1 (0.1)
AEN	1/MIN	AMBIENT	5	181 (5.5)	17.9 (1.4)	8.8 (0.2)		9.1 (0.5)
AFMR	0.01/MIN	60	10	149 (7.6)		7.5 (1.1)	9.89 (1.97)	7.80 (1.4)
AFNR	1/MIN	60	10	163 (3.4)		6.2 (1.4)	9.16 (3.08)	6.43 (1.8)
AFOR	10/MIN	60	10	164 (11)		6.8 (0.8)	11.23 (1.02)	7.12 (0.9)
AFM	0.01/MIN	AMBIENT	10	164 (6.2)	17.9 (1.4)	5.8 (1.90)	9.72 (1.76)	6.8 (1.2)
AFN	1/MIN	AMBIENT	10	178 (5.5)	20.0 (2.1)	1.7 (0.7)	5.67 (2.30)	4.0 (1.5)
AFO	10/MIN	AMBIENT	10	182 (10)	21.4 (3.4)	1.4 (0.4)	2.80 (1.48)	2.2 (1.3)
AFKS	0.001/MIN	-18	10	150 (10)		1.5 (0.2)	1.47 (0.39)	1.49 (0.3)
AFMS	0.01/MIN	-18	10	159 (14)		1.4 (0.2)	1.64 (0.99)	1.55 (0.6)
AFNS	10/MIN	-18	10	164 (10)		1.2 (.1)	1.28 (0.23)	1.25 (.1)
AFMU	0.01/MIN	-73	5	161 (28)		1.2 (0.2)	1.39 (0.32)	1.2 (0.2)

TEMPERATURES, AMBIENT, R=60°C, S=-18°C, U=-73°C
MATERIAL: AS4/3501-6; AEM & AEN are 2.54 cm X 15.24 cm; the rest are 0.64 cm X 3.05 cm

1.2 Strain Rate Effects on In-Plane Shear Properties

The initial plan for this study was to conduct low strain rates of 0.001-10/min using a screw-driven Instron, moderate strain rates of 100/min using a hydraulic MTS, and high strain rates of 1K-10K/min using a split-Hopkinson bar *[4]*. Unfortunately, due to budget cuts only the low strain rate tests were completed. The test matrix with results for AS4/3501-6 and AS4/APC-2 are provided in Tables 1 and 2, respectively. Test temperatures ranged from 60°C down to -73°C. Several experimental difficulties occurred during low-temperature testing. First, the rubber pads used to clamp the extensometer on the specimen became "glassy" which allowed for slippage whenever the specimen cracked. Second, the duration of the test was so short for the case of AS4/3501-6 that the computer could only collect about 10 data points (max rate is 50 points/second) prior to failure.

Figure 1 provides several sample stress-strain curves of the AS4/3501-6, $[\pm45]_{2S}$ tensile tests. We see that the initial elastic modulus remains virtually unchanged by the strain

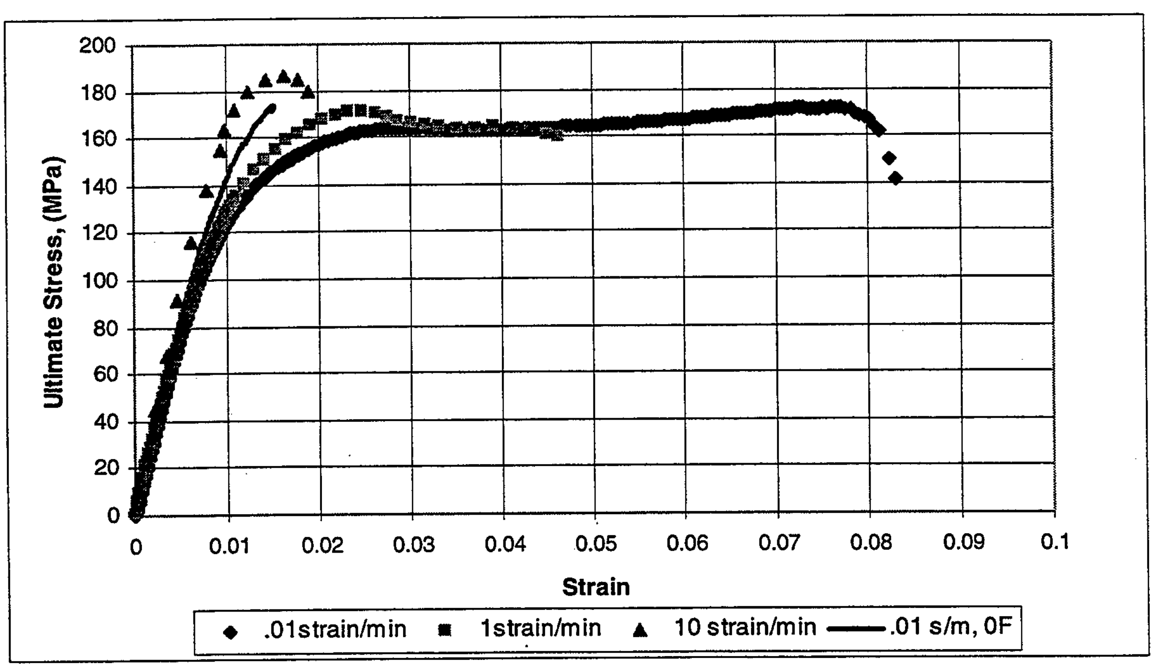

Figure 1. Comparison of Stress-Strain Curves for AS4/3501-6, $[\pm45]_{2S}$ Laminates.

TABLE 2
In-Plane Shear Properties of AS4/APC-2

SAMPLE	S. RATE	TEMP, C	NUMBER	ULT STRESS, MPa	YOUNG'S MODULUS, GPa	%STRAIN @ MAX LOAD	NORMALIZED WORK, MPa	ULTIMATE STRAIN
BEM	0.01/min	AMBIENT	6	232 (36.5)	17.9 (2.07)	11.6 (4.3)		11.6 (4.3)
BEN	1/min	AMBIENT	5	327 (11.7)	17.2 (1.38)	24.8 (7.7)		24.8 (7.7)
BFM	0.01/MIN	AMBIENT	9	280 (35.9)	17.9 (2.07)	15.8 (3.7)	32.7 (9.2)	16.1 (3.6)
BFN	1/MIN	AMBIENT	8	297 (38.6)	19.3 (2.76)	14.0 (4.2)	33.4 (10.3)	15.2 (4.1)
BFO	10/MIN	AMBIENT	8	290 (36.5)	17.9 (0.69)	13.9 (4.1)	32.6 (11.5)	14.6 (4.4)
BFMU	0.01/MIN	-73	10	270 (20.7)	19.3 (1.38)	11.0 (2)	24.2 (5.8)	11.0 (2)
BFNU	1/MIN	-73	10	268 (11.7)		8.7 (1.3)	20.3 (3.48)	8.9 (1.2)
BFOU	10/MIN	-73	10	276 (26.2)		8.3 (0.7)	21.4 (8.73)	8.5 (2.50)

TEMPERATURES, AMBIENT, R=60°C, S=-18°C, U=-73°C
MATERIAL: AS4/APC-2; BEM & BEN are 2.54 cm X 15.24 cm; the rest are 0.64 cm X 3.05 cm

rate, but the strain-to-failure is highly dependent on strain rate and temperature. The curve of one sample tested at 10/min and 20°C is similar to the curve for a second sample tested at 0.01/min and -18°C. The area under these curves was defined as the "normalized work to failure" for the purpose of comparison between tests. The idea proposed here is that test cases with higher work to failure should be more damage tolerant in a similar vane, as G_{Ic} and G_{IIc} are used as predictors of damage tolerance.

Figure 2 summarizes the test results obtained from AS4/3501-6. Keeping in mind that the standard deviation of the test results is rather high, we see several trends. The ultimate stress increases with strain rate at all three temperatures. The strain at maximum load and normalized work both decrease with strain rate at 20°C but don't show a meaningful trend at 60°C or -18°C. Perhaps the most surprising observation is that the strain at maximum load and normalized work drops nearly 75 percent when comparing data taken at 20°C and -18°C for a strain rate of 0.01/min. Figure 3 summarizes the test results obtained from AS4/APC-2. At 20°C we see essentially no trend with regard to strain rate. At -73°C there is a slight decrease in strain at maximum load and normalized work with increasing strain rate; however, the standard deviations are quite high.

Based on the limited test data developed thus far, it appears that the epoxy system, AS4/3501-6, which is known to have a lower toughness than the thermoplastic, AS4/APC-2, is much more sensitive to both temperature and strain rate. It is premature, however, to state that we can predict high strain rate performance by testing at moderate strain rates with reduced temperatures.

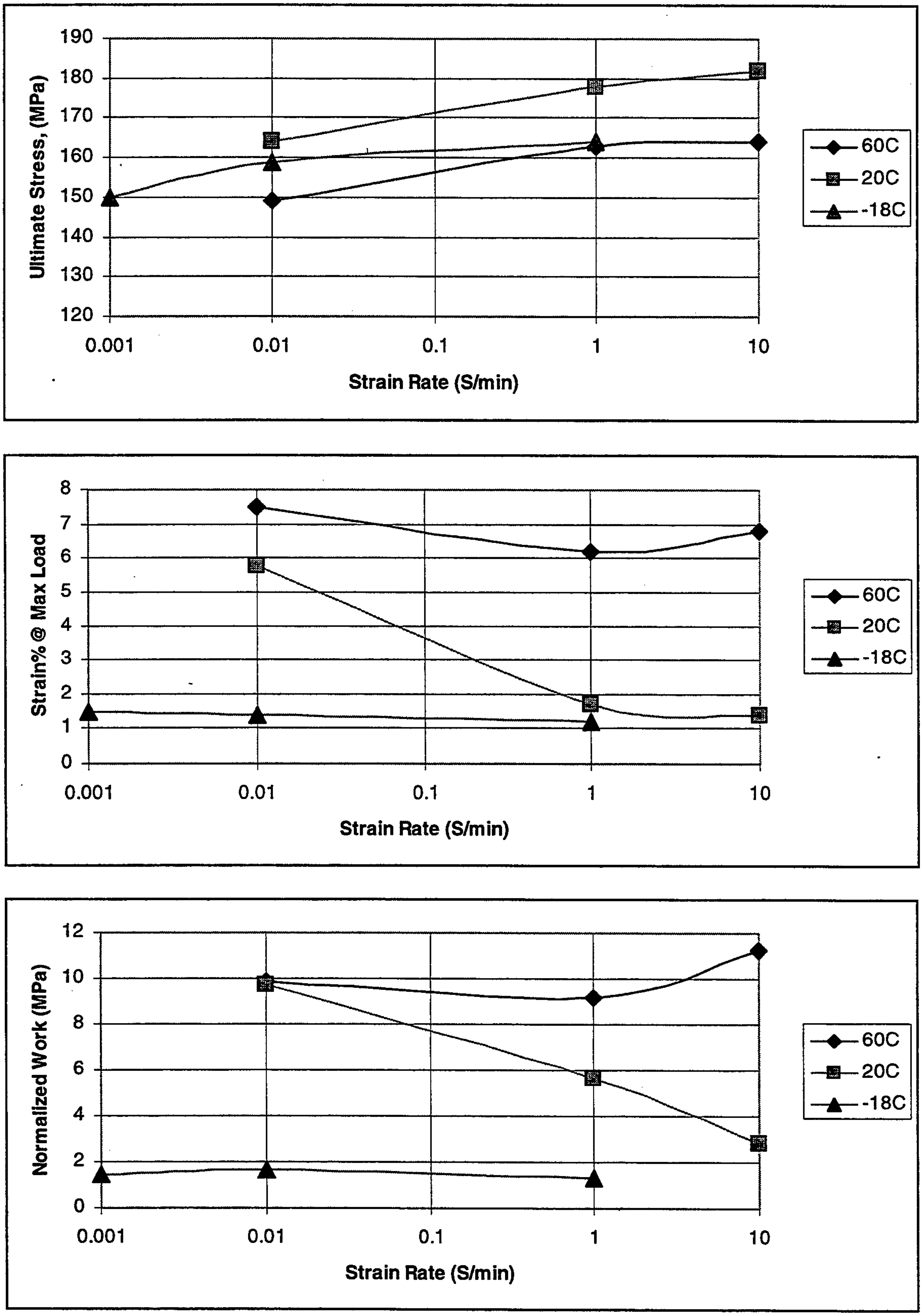

Figure 2. Tensile Data of AS4/3501-6, $[\pm45]_{2S}$ Laminates.

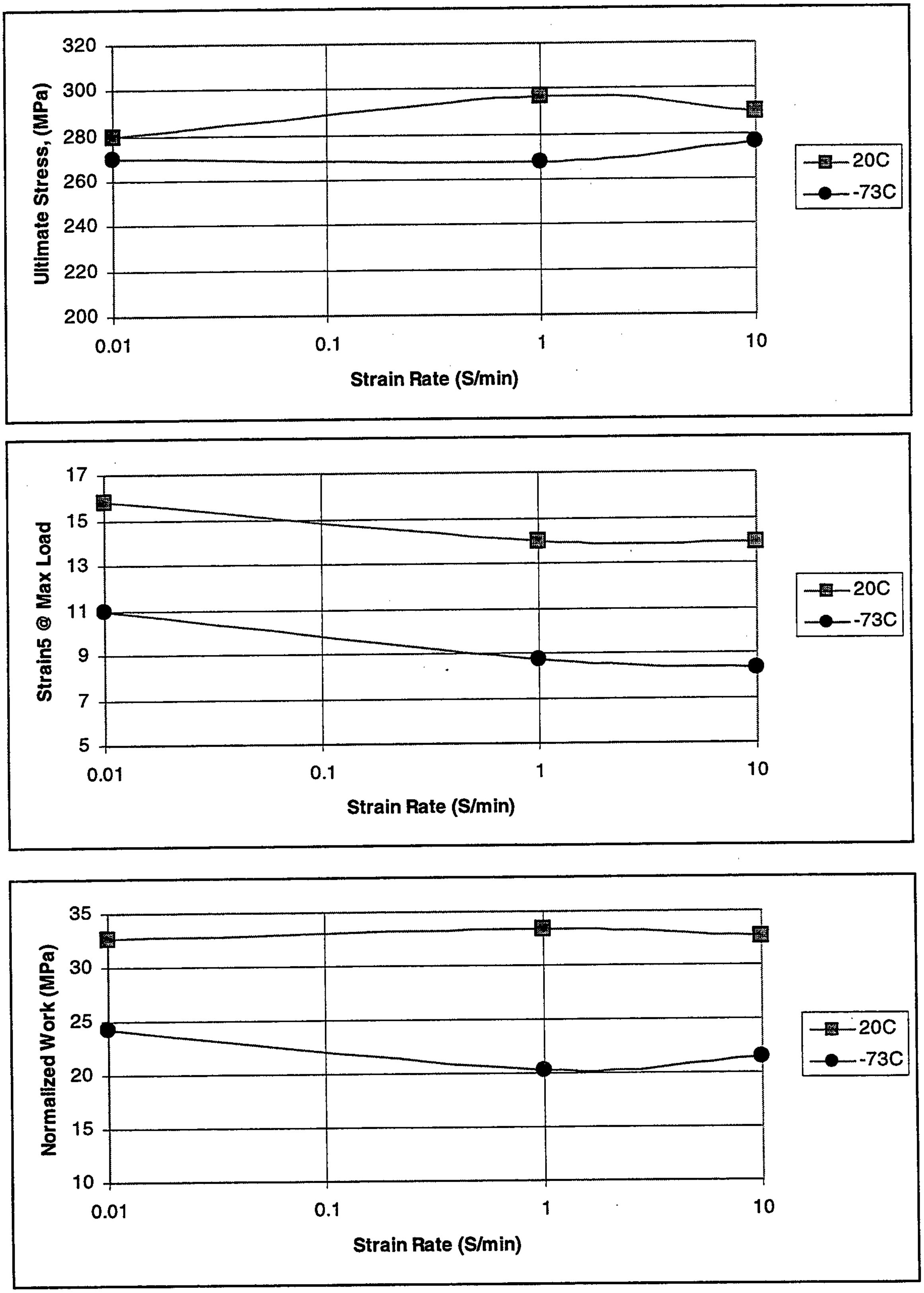

Figure 3. Tensile Data for AS4/APC-2, $[\pm 45]_{2S}$ Laminates.

2. INFRASTRUCTURE DEVELOPMENT FOR EVALUATING HIGH STRAIN RATE EFFECTS ON COMPOSITES

An effort was initiated to develop an in-house capability to conduct impact tests on composites as a means of characterizing novel toughening mechanisms such as nano-composites. The Rheometric's RDT-5000 impact tester was therefore resurrected. The unique feature of this drop-tower impact tester is that it has an environmental chamber which can control the temperature to ±100°C. As in the case of tensile testing where strain rate was limited, the impact velocity of this instrument is limited to approximately 10 m/sec, while a ballistic event is likely to occur at approximately 1000 m/sec. For this reason we hoped to conduct drop-tower impact tests at subambient temperatures to better understand the material's response during ballistic tests.

A test plan was developed to compare impact damage on several composite materials using both techniques. Table 3 lists the composite laminates which were fabricated to conduct this study. Unfortunately, due to the budget cuts none of these laminates have been tested to date.

TABLE 3
Composite Laminates Fabricated for Impact Tests

AS4/ 3501-6	
$[0/45/90/-45]_S$, 12" x 12"	4 panels
$[0/45/90/-45]_{2S}$, 12" x 12"	4 panels
$[0/45/90/-45]_{4S}$, 12" x 12"	4 panels
AS4/APC-2	
$[0/45/90/-45]_S$, 12" x 12"	4 panels
$[0/45/90/-45]_{2S}$, 12" x 12"	4 panels
$[0/45/90/-45]_{4S}$, 12" x 12"	4 panels
T300/LTM-45	
$[0/45/90/-45]_S$, 12" x 12"	8 panels
$[0/45/90/-45]_{2S}$, 12" x 12"	12 panels
$[0/45/90/-45]_{4S}$, 12" x 12"	12 panels
T650-42/RADEL 8320	
$[0/45/90/-45]_S$, 12" x 12"	8 panels
$[0/45/90/-45]_{2S}$, 12" x 12"	8 panels
$[0/45/90/-45]_{4S}$, 12" x 12"	8 panels

3. TECHNICAL SUPPORT ACTIVITIES FOR NANOCOMPOSITE PROCESSING AND CHARACTERIZATION

Characterization experiments were conducted on several formulations of nano-composite materials in support of research conducted by an Air Force visiting scientist.

A total of 33 DMA scans were conducted in support of the nanocomposite formulation studies.

A total of 45 samples were run on the thermal analysis equipment during this period. They include 828/D230, 828/D2000, 828/D400, 828/clay, 828/Jeffamine/clay, and Closite S30. Different amounts of additives were used in each case. A number of specimens were also examined using transmission electron microscopy.

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