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ULTRASONIC SHEAR WAVE VELOCITY IN CLF/CMT GRAPHITE
FROM ROOM TEMPERATURE TO 2000° F

John H. Gieske

Prepared by Sandia National Laboratories, Albuquerque,
New Mexico 87185 and Livermore, California 94550 for
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FROM ROOM TEMPERATURE TO 2000°F

John H. Gieske
Nondestructive Testing Technology Division 1552
Sandia National Laboratories
Albuquerque, New Mexico 87185

ABSTRACT

The temperature dependence of the ultrasonic shear velocity in CLF/CMT graphite was determined from room temperature to 2000°F using a pulse-echo technique. Data are presented for five 0.75 inch diameter specimens all machined from the same CLF/CMT billet. Plots of ultrasonic pulse-echo radial and axial scans of the billet which characterize the material property uniformity of the billet are also given.

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I. Introduction

The purpose of this work is to supplement the previously reported ultrasonic shear velocity measurements for CMT graphite from room temperature to 6500°F¹. For the measurements to 6500°F, the velocity data from room temperature to 2000°F were lacking due to the temperature measuring technique of the 6500°F furnace.

The temperature dependence of the ultrasonic shear velocity from room temperature to 6500°F is used for the calculation of the ablation of a CMT nose tip of a reentry vehicle as measured by an ultrasonic recession gage. Discrepancies in the calculated real time recession by the ultrasonic gage compared with an independently measured recession in laboratory test models, pointed out that a detailed knowledge of the temperature dependence of the ultrasonic shear velocity from room temperature to 2000°F was necessary.

II. Ultrasonic Characterization of the CLF/CMT Billet

A computerized ultrasonic pulse-echo characterization of the billet was performed prior to fabrication of the test specimens in order to assess the quality of the billet as well as to determine from which areas of the billet to machine specimens for optimum test performance. The complete pulse-echo flaw data, velocity contours, and relative attenuation plots for the billet are shown in Appendix A.

A review of the data in Appendix A indicates that the billet is of good quality. The reflection planes appearing in the axial direction as seen in the first flaw characterization plot in Appendix A are normally observed for a CMT billet. The variation of three to four percent in the longitudinal velocity measured in the axial and radial directions across the

volume of the billet is observed for all CMT billets. The 6 db relative amplitude variation in the radial direction is also normal for a CMT billet. No defects were observed in the billet which should be avoided in machining the 0.75 inch diameter test specimens. The location of each test specimen as it was machined from the billet is shown schematically in Figure A2.

III. Experimental Methods

The ultrasonic technique used for the determination of the shear wave velocity as a function of temperature is the same one used previously^{1,2}. A 0.5 MHz shear transducer was coupled to one end of an indexed specimen in the form of a 0.75 inch diameter rod approximately 8 inches long. The indexed section, which provided a one inch gage length for the measurement, was placed in the furnace and the transducer end of the specimen was threaded into a water-cooled brass holder to prevent excessive heating of the transducer. The specimen was machined with 32 threads per inch over the entire length of the specimen to reduce trailing or mode converted wave interference with the echoes of interest. The shear transducer was 1 inch in diameter and was coupled to the specimen using the uncured resin from Hysol Epoxy-Patch #9340.

The furnace was a 2000°F resistance heating element furnace with a 2 inch diameter bore. The furnace was operated in the vertical position where a constant flow of helium gas was introduced into the top of the furnace which was sealed from the atmosphere. This assured minimum oxidation of the test specimen at the elevated temperatures.

The indexed section of each specimen was made by milling a slot in the side of the specimen to provide a 1.000 inch gage length from the slot to the

end of the specimen. The slot was machined deep enough so that the reflection from the slot and the end of the rod was approximately equal. Test specimens #2, #3, and #9 were machined with the indexed section at the end of the specimen which corresponded to the lot numbered end of the billet. Test specimens #5 and #8 were machined with the index section at the end of the specimen corresponding to the opposite end of the billet. This was done to exhibit any temperature dependent properties of the billet which might exist from one end of the billet to the other.

The ultrasonic velocity is calculated at each temperature by measuring the delay time difference between the pair of echoes from the slot and the end of the specimen. The velocity is given by

$$V = 2L/t \quad (1)$$

where t is the measured delay time and L is the indexed length of the specimen from the slot to the end of the specimen.

The length L at temperature was corrected for thermal expansion using the thermal expansion data³ expressed by

$$\begin{aligned} (L - L_0)/L_0 = & - .127673E-3 + .172325E-5 T + 8.74177E-10 T^2 \\ & - 2.75686E-13 T^3 + 4.77364E-17 T^4 - 2.39801E-21 T^5 \end{aligned} \quad (2)$$

where T is degrees Fahrenheit and L_0 is the room temperature length. The test temperature was read with a Chromel-Alumel thermocouple placed against the indexed section of the test specimen.

The delay time measurement t between the pair of echoes from the slot and the end of the specimen at each temperature was accomplished with the electronic apparatus shown schematically in Figure 1. At each temperature determination, the ultrasonic waveform which included the pair of echoes of

interest was recorded on a X-Y plotter by using the boxcar integrator. The X direction of the plotter was calibrated for two microseconds per centimeter with the use of two adjustable start and stop time gates set exactly 30.00 μ seconds apart. The signal of the two gates is recorded on each plot of the ultrasonic waveforms.

The ultrasonic waveform at each temperature determination was plotted on the same graph where the thermocouple temperature and time of day was recorded next to the waveform. The delay time measurement, t , used in Equation (1) between the pair of echoes of interest from one waveforms to the next can be calculated directly from the graph. Each delay time measurement t can be determined from the graph with an accuracy of ± 0.1 μ second which represents an error of no greater than 0.35% in total delay time.

IV. Test Results

The ultrasonic waveforms recorded at approximately 100°F increments for five test specimens from room temperature to 2000°F are shown in Appendix B. Tables 1 through 5 list the temperature, the corrected index length, the measured delay time as read from the graphs of Appendix B, and the calculated shear wave velocity using Equation (1). The normalized velocity value at each temperature is also given where the room temperature velocity value for each specimen was taken as the normalizing velocity.

For completeness, decreasing temperature data were also recorded. The decreasing temperature data, however, were accelerated to fit the test run within the normal working day. Due to the rapid decrease in temperature, thermal equilibrium may not have occurred at all temperatures.

However, a slower decrease in temperatures was conducted for specimens #5 and #8. No significant change in the temperature dependence of the shear wave velocity was observed on decreasing temperature.

The shear wave velocity versus temperature is plotted in Figure 2 for all five test specimens. The spread in the data represents the three to four percent difference in the absolute wave velocity between the five specimens. The temperature dependence of the wave velocity for the five specimens agrees to approximately 1.5% which can be seen in Figure 3 where a plot of the normalized velocity versus temperature is given for each specimen.

V. Summary and Conclusions

The ultrasonic shear wave velocity was measured in CLF/CMT graphite from room temperature to 2000°F in 100°F increments. Shear wave velocities were measured for five specimens machined from the same CMT billet where an agreement of 3.5 percent was observed between the specimens over the entire temperature range. The temperature dependence of the normalized shear wave velocity agreed to within 1.5 percent for the five specimens over the entire temperature range.

FIGURE 1. SCHEMATIC DIAGRAM OF THE ELECTRONIC APPARATUS USED FOR RECORDING THE ULTRASONIC WAVE TRACE AND MEASURING THE TIME DELAY DIFFERENCE BETWEEN TWO POINTS OF THE WAVE TRACE.

FIGURE 2. SHEAR WAVE VELOCITY VERSUS TEMPERATURE FOR FIVE SPECIMENS FROM BILLET 154J-04

FIGURE 3. NORMALIZED SHEAR VELOCITY VERSUS TEMPERATURE

TABLE 1 - SHEAR VELOCITY DATA FOR CMT GRAPHITE SPECIMEN #2

<u>DEGREE</u> <u>F</u>	<u>LENGTH</u> <u>MM</u>	<u>DELAY</u> <u>MICROSEC</u>	<u>VELOCITY</u> <u>MM/MICROSEC</u>	<u>NORMALIZED</u> <u>V/VO</u>
77	50.801	31.85	1.595	1
198	50.812	31.75	1.6	1.003
300	50.823	31.85	1.596	1
401	50.835	31.75	1.601	1.004
501	50.847	31.85	1.596	1.001
605	50.86	31.9	1.594	1
700	50.872	31.75	1.602	1.005
802	50.886	31.5	1.615	1.013
901	50.9	31.6	1.611	1.01
1000	50.914	31.6	1.611	1.01
1100	50.928	31.6	1.612	1.01
1200	50.943	31.65	1.61	1.009
1302	50.958	31.45	1.62	1.016
1404	50.974	31.4	1.623	1.018
1501	50.989	31.35	1.626	1.02
1629	51.009	31.15	1.638	1.027
1721	51.023	31.1	1.641	1.029
1805	51.037	31.05	1.644	1.031
1906	51.053	30.85	1.655	1.038
2001	51.069	30.65	1.666	1.045
2023	51.072	30.55	1.672	1.048
2028	51.073	30.6	1.669	1.046
2000	51.069	30.5	1.674	1.05
1900	51.052	30.5	1.674	1.049
1800	51.036	30.6	1.668	1.046
1700	51.02	30.65	1.665	1.044
1600	51.004	30.8	1.656	1.038
1500	50.989	30.9	1.65	1.035
1400	50.973	31	1.644	1.031
1300	50.958	31.05	1.641	1.029
1200	50.943	31.1	1.638	1.027
1100	50.928	31.2	1.632	1.023
1000	50.914	31.3	1.627	1.02
900	50.899	31.35	1.624	1.018
800	50.886	31.45	1.618	1.014
700	50.872	31.55	1.612	1.011
600	50.859	31.6	1.609	1.009
500	50.847	31.65	1.607	1.007
83	50.801	31.9	1.593	.998

TABLE 2 - SHEAR VELOCITY DATA FOR CMT GRAPHITE SPECIMEN #3

DEGREE F	LENGTH MM	DELAY MICROSEC	VELOCITY MM/MICROSEC	NORMALIZED V/VO
89	50.802	31.95	1.59	1
224	50.815	32	1.588	.999
301	50.824	32.05	1.586	.997
410	50.836	32.05	1.586	.998
523	50.85	31.85	1.597	1.004
599	50.859	31.8	1.599	1.006
690	50.871	31.7	1.605	1.009
797	50.885	31.55	1.613	1.014
900	50.899	31.55	1.613	1.015
1002	50.914	31.4	1.621	1.02
1101	50.928	31.35	1.625	1.022
1200	50.943	31.3	1.628	1.024
1299	50.958	31.25	1.631	1.026
1401	50.973	31.15	1.636	1.029
1496	50.928	31.05	1.642	1.033
1624	51.008	30.8	1.656	1.042
1723	51.024	30.8	1.657	1.042
1801	51.036	30.7	1.662	1.046
1900	51.052	30.6	1.668	1.049
1950	51.06	30.5	1.674	1.053
1900	51.052	30.5	1.674	1.053
1800	51.036	30.6	1.668	1.049
1700	51.02	30.55	1.67	1.05
1600	51.004	30.8	1.656	1.041
1500	50.989	30.7	1.661	1.045
1410	50.975	30.9	1.65	1.038
1312	50.96	30.95	1.647	1.036
1208	50.944	31	1.643	1.034
1117	50.931	31.15	1.635	1.028
1025	50.917	31.1	1.637	1.03
912	50.901	31.25	1.629	1.024
790	50.884	31.3	1.626	1.022
692	50.871	31.55	1.612	1.014
593	50.858	31.65	1.607	1.011
506	50.847	31.6	1.609	1.012
391	50.834	31.7	1.604	1.009
287	50.822	31.6	1.608	1.012
198	50.812	31.8	1.598	1.005
92	50.802	31.8	1.598	1.005
92	50.802	32.1	1.583	.995

TABLE 3 - SHEAR VELOCITY DATA FOR CMT GRAPHITE SPECIMEN #5

DEGREE F	LENGTH MM	DELAY MICROSEC	VELOCITY MM/MICROSEC	NORMALIZED V/VO
79	50.801	32	1.588	1
205	50.813	32.15	1.581	.995
302	50.824	32.2	1.578	.994
403	50.835	32.1	1.584	.997
498	50.847	32.1	1.584	.997
605	50.86	31.95	1.592	1.002
697	50.872	31.9	1.595	1.004
803	50.886	31.8	1.6	1.008
900	50.899	31.85	1.598	1.006
1003	50.914	31.6	1.611	1.015
1100	50.928	31.6	1.612	1.015
1204	50.943	31.55	1.615	1.017
1301	50.958	31.5	1.618	1.019
1401	50.973	31.35	1.626	1.024
1501	50.929	31.25	1.632	1.027
1653	51.013	31.1	1.64	1.033
1718	51.023	31	1.646	1.036
1801	51.036	30.8	1.657	1.043
1900	51.052	30.85	1.655	1.042
1993	51.067	30.8	1.658	1.044
1899	51.052	30.75	1.66	1.045
1799	51.036	30.8	1.657	1.043
1689	51.018	30.8	1.656	1.043
1601	51.004	30.9	1.651	1.039
1494	50.988	31.05	1.642	1.034
1397	50.973	31.1	1.639	1.032
1299	50.958	31.1	1.639	1.032
1196	50.942	31.2	1.633	1.028
1102	50.928	31.35	1.625	1.023
996	50.913	31.4	1.621	1.021
907	50.9	31.5	1.616	1.018
698	50.872	31.75	1.602	1.009
506	50.847	31.7	1.604	1.01
336	50.827	31.9	1.593	1.003
86	50.801	32.05	1.585	.998

TABLE 4 - SHEAR VELOCITY DATA FOR CMT GRAPHITE SPECIMEN #8

DEGREE F	LENGTH MM	DELAY MICROSEC	VELOCITY MM/MICROSEC	NORMALIZED V/V0
76	50.8	32.3	1.573	1
209	50.814	32.4	1.568	.997
335	50.827	32.45	1.566	.996
422	50.837	32.4	1.569	.997
500	50.847	32.45	1.567	.996
604	50.86	32.3	1.575	1.001
700	50.872	32.15	1.582	1.006
801	50.886	32.05	1.588	1.009
903	50.9	32.05	1.588	1.01
1001	50.914	31.95	1.594	1.013
1100	50.928	31.9	1.596	1.015
1200	50.943	31.95	1.594	1.014
1301	50.958	31.85	1.6	1.017
1401	50.973	31.75	1.605	1.021
1500	50.929	31.69	1.611	1.024
1600	51.004	31.6	1.614	1.026
1708	51.021	31.55	1.617	1.028
1826	51.04	31.4	1.625	1.033
1900	51.052	31.25	1.634	1.039
2000	51.069	31.05	1.645	1.046
2002	51.069	31.05	1.645	1.046
1897	51.052	31.05	1.644	1.045
1804	51.037	31.05	1.644	1.045
1694	51.019	31.15	1.638	1.041
1593	51.003	31.2	1.635	1.039
1496	50.988	31.3	1.629	1.036
1404	50.974	31.4	1.623	1.032
1304	50.958	31.45	1.62	1.03
1207	50.944	31.55	1.615	1.027
1101	50.928	31.65	1.609	1.023
1000	50.914	31.75	1.604	1.019
888	50.898	31.9	1.596	1.014
819	50.888	31.95	1.593	1.013
81	50.801	32.5	1.563	.994

TABLE 5 - SHEAR VELOCITY DATA FOR CMT GRAPHITE SPECIMEN #9

<u>DEGREE</u> <u>F</u>	<u>LENGTH</u> <u>MM</u>	<u>DELAY</u> <u>MICROSEC</u>	<u>VELOCITY</u> <u>MM/MICROSEC</u>	<u>NORMALIZED</u> <u>V/VO</u>
76	50.8	31.45	1.615	1
218	50.815	31.6	1.608	.996
301	50.824	31.6	1.608	.996
405	50.835	31.7	1.604	.993
501	50.847	31.6	1.609	.996
601	50.859	31.55	1.612	.998
700	50.872	31.45	1.618	1.002
800	50.886	31.3	1.626	1.007
901	50.9	31.35	1.624	1.005
1000	50.914	31.25	1.629	1.009
1100	50.928	31.3	1.627	1.007
1200	50.943	31.25	1.63	1.009
1300	50.958	31.2	1.633	1.011
1401	50.973	31.05	1.642	1.017
1500	50.989	30.95	1.647	1.02
1612	51.006	30.75	1.659	1.027
1702	51.02	30.55	1.67	1.034
1800	51.036	30.45	1.676	1.038
1817	51.039	30.35	1.682	1.041
1794	51.035	30.4	1.679	1.039
1700	51.02	30.45	1.676	1.037
1600	51.004	30.55	1.67	1.034
1500	50.989	30.6	1.666	1.032
1400	50.973	30.65	1.663	1.03
1300	50.958	30.7	1.66	1.028
1200	50.943	30.8	1.654	1.024
1100	50.928	30.85	1.651	1.022
1000	50.914	30.9	1.648	1.02
900	50.899	30.95	1.645	1.018
800	50.886	31.1	1.636	1.03
700	50.872	31.2	1.631	1.01
600	50.859	31.2	1.63	1.009
500	50.847	31.25	1.627	1.007
400	50.835	31.35	1.622	1.004
300	50.823	31.45	1.616	1.001
264	50.819	31.35	1.621	1.004
81	50.801	31.85	1.595	.988

APPENDIX A

Computerized Pulse Echo Ultrasonic Characterization of CMT Billet 154J-04

TEST#CMTEE1
 DATE 5 20 80
 ECHOES PLOTTED
 WITH AMPLITUDES
 IN THE RANGE
 -15 DB TO 0 DB AMPLITUDE
 DIAMETER= 3 INCHES
 THICKNESS= 8.125 INCHES

DATA BETWEEN DEPTH OF
 .42 INCHES TO 7.982 INCHES

TOP VIEW

DEPTH VIEW

NUMBERED END

NUMBERED END

FIGURE A1. Pulse Echo Flaw Data resulting from scanning obverse (# end) and reverse end of the billet with a 1.0 MHz 1/2 inch transducer. Black areas show small amplitude reflections from planes of abrupt material property variations which are not detrimental to the integrity of the billet but may distort wave velocity measurement.

TEST#CMTEE1
 DATE 5 20 88
 ECHOES PLOTTED
 WITH AMPLITUDES
 IN THE RANGE
 -15 DB TO 0 DB AMPLITUDE
 DIAMETER= 3 INCHES
 THICKNESS= 8.125 INCHES

VELOCITY CONTOUR

.083	A inches/ μ second
.084	B
.085	C

FIGURE A2. (a) Pulse Echo ultrasonic longitudinal velocity contour measurement in Longitudinal Velocity in Axial Direction (across grain) of the axial direction of the billet. (b) Approximate locations in billet from which 3/4 inch diameter test specimens were machined.

TEST#CMTA1
DATE 5 13 80
ECHOES PLOTTED
WITH AMPLITUDES
IN THE RANGE
-15 DB TO 0 DB

FIGURE A3. Ultrasonic pulse echo relative attenuation in the radial direction of the billet. Large variations of the ultrasonic echo amplitude are recorded but are not unusual for a CMT billet. Black shows locations where the signal amplitude was at least 6 db above the signal at the white areas. Numbered end of billet is at the bottom of the plot.

TEST#CMTU1
 DATE 5 14 80
 ECHOES PLOTTED
 WITH AMPLITUDES
 IN THE RANGE
 -15 DB TO 0 DB

VELOCITY LABEL
 ? .106 A

FIGURE A1. Ultrasonic longitudinal velocity values measured in the radial direction of the billet. The numbered end of the billet is at the bottom of the plot. A = .106 in./ μ sec; B = .104-.105 in./ μ sec; C = .107-.108 in./ μ sec.

TEST#LHCMT1
 DATE 5 13 80
 ECHOES PLOTTED
 WITH AMPLITUDES
 IN THE RANGE
 -9 DB TO 0 DB
 SECTIONS OF
 CYLINDER AT
 EVERY 45 DEGREES

DEPTH VIEW TO
 THE CENTER OF THE
 CYLINDER SHOWN
 TO RIGHT OF
 TOP VIEW

FIGURE A5. Pulse Echo Flaw Data in the radial direction of the billet. No flaws of consequence were recorded indicating a billet of good quality.

APPENDIX B

Recorded Waveforms for CMT Specimens #2, #3, #5, #8, and #9 as a Function
of Increasing and Decreasing Temperature in Degrees Fahrenheit

30 μ sec. 2:31 7/2/80

2023 °F 2:30
 2001.5 °F 2:15
 1906.3 °F 1:25
 1805.3 °F 12:53
 1721.2 °F 12:30
 1629.2 °F 11:50
 1501.5 °F 11:25
 1404.7 °F 11:10
 1302.5 °F 10:56
 1200.8 °F 10:43
 1100.5 °F 10:28
 1000.6 °F 10:15
 901.3 °F 10:00
 802.9 °F 9:49
 700.5 °F 9:39
 605.4 °F 9:28
 501.9 °F 9:18
 401.4 °F 9:08
 300.8 °F 8:57
 198.7 °F 8:35
 77.3 °F 7:57

CMT
 SPECIMEN #2
 30 μ sec. 7/2/80

FIGURE B1. Pulse Echo Waveforms showing echoes from gage section of 1.000 inch between milled slot and end of specimen #2 for increasing temperatures.

FIGURE B2. Pulse Echo Waveforms showing echoes from gage section of 1.000 inch between milled slot and end of specimen #2 for decreasing temperatures.

FIGURE B3. Pulse Echo Waveforms showing echoes from gage section of 1.000 inch between milled slot and end of specimen #3 for increasing temperatures.

FIGURE B4. Pulse Echo Waveforms showing echoes from gage section of 1.000 inch between milled slot and end of specimen #3 for decreasing temperatures.

30 μ sec. CALIB. 2:02 7/9/80

20	1993.6°F	2:00
19	1900.8°F	1:17
18	1801.0°F	12:49
17	1718.9°F	12:31
16	1653.4°F	11:50
15	1501.3°F	11:26
14	1401.2°F	11:13
13	1301.9°F	11:00
12	1204.8°F	10:48
11	1100.6°F	10:34
10	1003.2°F	10:16
9	900.6°F	9:58
8	803.1°F	9:45
7	697.8°F	9:30
6	605.5°F	9:14
5	498.7°F	9:00
4	403.0°F	8:40
3	302.2°F	8:25
2	205.5°F	8:10
1	79.6°F	7:55

CMT Graphite #5
SPECIMEN 5
30 μ sec. 7/9/80

FIGURE B5. Pulse Echo Waveforms showing echoes from gage section of 1.000 inch between milled slot and end of specimen #5 for increasing temperatures.

FIGURE B6. Pulse Echo Waveforms showing echoes from gage section of 1.000 inch between milled slot and end of specimen #5 for decreasing temperatures.

FIGURE B7. Pulse Echo Waveforms showing echoes from gage section of 1.000 inch between milled slot and end of specimen #8 for increasing temperatures.

30 μ sec. CALIB. 7/8/80

81.1°F 8:20 7/8/80

819.7°F 4:28

888.4°F 4:14

1000.3°F 3:57

1101.0°F 3:43

1207.3°F 3:18

1304.5°F 3:00

1404.5°F 2:47

1496.8°F 2:26

1593.3°F 2:14

1694.5°F 2:03

1804.7°F 1:49

1897.1°F 1:35

2002°F 1:26

CMT

SPECIMEN #8 7/7/80

30 μ sec. CALIB.

FIGURE B8. Pulse Echo Waveforms showing echoes from gage section of 1.000 inch between milled slot and end of specimen #8 for decreasing temperatures.

FIGURE B9. Pulse Echo Waveforms showing echoes from gage section of 1.000 inch between milled slot and end of specimen #9 for increasing temperatures.

FIGURE B10. Pulse Echo Waveforms showing echoes from gage section of 1.000 inch between milled slot and end of specimen #9 for decreasing temperatures.

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