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ULTRASONIC MEASUREMENT OF ELASTIC MODULI OF 17-4 PH STAINLESS
STEEL AND URANIUM - 2 MOLYBDENUM FROM -40°C TO 800°C

MASTER

John H. Gieske

TRANSPORTATION TECHNOLOGY CENTER

Prepared by Sandia Laboratories, Albuquerque, New Mexico 87185
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STEEL AND URANIUM - 2 MOLYBDENUM FROM -40°C TO 800°C

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ABSTRACT

Young's Modulus, shear modulus, and Poisson's ratio for 17-4 PH stainless steel and Uranium - 2 Molybdenum are calculated from ultrasonic longitudinal and shear velocities determined from -40°C to 800°C. The ultrasonic velocities were determined at elevated temperatures using a through-transmission buffer rod arrangement. An indium-gallium slurry bond was used as an ultrasonic couplant between Cupernickel 10 alloy buffer rods and the specimen. Microstructural changes and phase transitions in the specimens are evident from the temperature dependence of the ultrasonic data.

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I. Introduction

Both 17-4 PH stainless steel and Uranium - 2 (wt. pct.) Molybdenum are being considered for use in Liquid Metal Breeder Reactor spent fuel shipping containers and other nuclear materials transportation systems. The structural assessment of such systems under normal operating temperature conditions and extreme temperature excursions due to possible accident scenarios requires knowledge of the thermal-elastic properties of these materials from -40°C to 800°C . This report provides Young's modulus, shear modulus, and Poisson's ratio as calculated from ultrasonic longitudinal and shear velocity data from -40°C to 800°C .

II. Ultrasonic Technique

Basic ultrasonic travel time data were obtained on specimens with flat and parallel sides of dimensions 6.35 mm x 9.525 mm x 12.7 mm. Ten MHz transducers were used for the longitudinal wave measurements and five MHz transducers were used for the shear wave measurements. Precise longitudinal and shear data were taken at 25°C by the pulse-echo overlap (PEO) method¹ using a Panametrics 5053 Ultrasonic Time Intervalometer². Using the same apparatus, longitudinal and shear data was obtained from 25°C to -40°C using the PEO method in the through-transmission mode³. Non-aq stopcock grease was used as the couplant between transducers and the specimen at low temperatures.

The travel time measurements at elevated temperatures were obtained using a two buffer rod through-transmission method⁴. The travel time through the specimen at elevated temperatures is obtained by subtracting $1/2$ the pulse-echo travel time of each buffer rod from the through-transmission travel time of the buffer rod-specimen-buffer rod string. An

indium-gallium slurry bond was used as the ultrasonic couplant between Cupronickel 10 alloy buffer rods and the specimen. The indium-gallium mixture was rubbed onto the faces of the specimen and buffer rods with a wooden stick until good wetting of the surfaces occurred. Details of the two buffer rod-through-transmission methods and experimental apparatus are given in Reference 4.

One advantage of the through-transmission buffer rod method over the single buffer rod pulse-echo method is that travel time data can be obtained with either poor bond coupling efficiency between the buffer rods and the specimen or with high specimen attenuation of the ultrasonic waves. This is important since high specimen attenuation is common for most materials at elevated temperatures. In addition, wave interferences with trailing pulses due to mode conversions in the buffer rods are not a problem with the through-transmission method as it is with the pulse-echo method.

Specimen oxidation above 25°C was minimized by purging the heating furnace with argon gas. The temperature of the specimen was read with a chromel/Alumel thermocouple calibrated to $\pm 0.1^\circ\text{C}$. Travel time measurements could be made to ± 0.01 μsecond . This results in calculated velocity errors of better than ± 2 percent.

III. Moduli Calculations

The ultrasonic velocities at temperature are obtained by measuring the travel time for the ultrasonic wave to propagate through a known specimen length. The velocity is then determined by

$$V = \frac{L}{t} \quad (1)$$

where L is the specimen length at temperature and t is the measured travel time. The length L is corrected from the room temperature value L_R using thermal expansion data. The elastic moduli are calculated from the longitudinal velocity V_L and the shear velocity V_S by the equations

$$G = \rho V_S^2 \quad (2)$$

$$E = 2G(1 + \sigma) \quad (3)$$

and

$$\sigma = \frac{\left[1 - \frac{1}{2} V_L^2 / V_S^2 \right]}{\left[1 - V_L^2 / V_S^2 \right]} \quad (4)$$

where G , E , σ , and ρ are the shear modulus, Young's modulus, Poisson's ratio, and density respectively. The density ρ is calculated from the room temperature length L_R and the temperature corrected length L by

$$\rho = \rho_R \left(L_R / L \right)^3. \quad (5)$$

IV. Experimental Results

The experimental data were obtained in three steps. Initially longitudinal and shear velocities were determined at 25°C for two specimens of each material. Measurements were made in all three dimensions of the two specimens. Isotropy of the specimens was assured since velocities agreed to better than 0.5% for all measurements. Second, the low temperature determinations were made using the 25°C travel time value as a reference where changes from the 25°C value down to -40°C were recorded. Lastly,

the elevated temperature determinations were also made by measuring the change of the travel time from the 25°C value as a reference. Approximately 10 minutes were allowed at each temperature before ultrasonic data was obtained. The longitudinal data and shear data were obtained at elevated temperatures in two separate heating cycles.

The velocities and moduli values for the two separate temperature tests are listed in Table 1 for the 17-4 PH SS and in Table 2 for the U - 2 Mo alloy. The elevated temperature data is listed in the order the data was recorded. Specimen length and density corrections were made using thermal expansion data of Reference 5.

Plots of the longitudinal and shear velocity, Young's modulus, shear modulus, Poisson's ratio as a function of temperature for the 17-4 PH SS are seen in Figures 1, 2, 3, and 4. Corresponding plots of the velocity and moduli for the U - 2 Mo specimens are seen in Figures 5, 6, 7, and 8. A least-squares polynomial fit to the moduli was made to fourth degree. The coefficients calculated are listed in Table 3 for both materials.

V. Discussion of Experimental Results

From the plots of the ultrasonic velocity and elastic moduli versus temperature for both the 17-4 PH SS and the U - 2 Mo several abrupt changes in the slope of the curves are noted. These changes in slope can be explained in most part by microstructural changes in the material as a function of temperature. Changes in the elastic moduli usually accompany changes of phase where abrupt deviations of the ultrasonic velocity and attenuation are observed. Furthermore, subtle changes in the ultrasonic velocity and attenuation versus temperature have been used to study phase changes in some materials⁶. Conversely, explanations of anomalous ultrasonic velocity and

attenuation values can be obtained from phase diagrams.

17-4 PH Stainless Steel

In Figure 1, the ultrasonic and shear velocities exhibit a linear function of temperature from -40°C to approximately 500°C . From 500°C to the highest temperature measured (889°C), a second linear relationship of increased slope is displayed by the data. It is noted that 17-4 PH is a metastable precipitation hardening stainless steel strengthened by precipitation of Cu rich particles. The material was initially aged for 4 hours at 650°C and is considered to be in the overaged condition where no further Cu precipitation is taking place. Therefore the most likely explanation for the slope change in velocity at 500°C is formation of reverted austenite.

In Figure 3, a variation of 3-5% in shear modulus is noted at 250°C to 350°C between the increasing and decreasing temperature data. This variation is believed to be due to experimental error introduced because of poor coupling efficiency between the buffer rods and the specimen. The shear wave propagating through the buffer rod-specimen string was extremely low in amplitude at the beginning of the test. On increasing temperature the amplitude of the shear wave increased substantially from 400°C to 500°C . On decreasing temperature, a very small amplitude signal was again observed at 250°C which made accurate travel time measurements difficult.

U - 2 Mo

The experimental travel time data for determination of the ultrasonic velocity for the Uranium - 2 Molybdenum specimen exhibited a number of abrupt changes as a function of temperature. Figures 9 and 10 show the normalized travel time measured with two U - 2 Mo specimens as a function of increasing and decreasing temperatures for the longitudinal and shear waves. Two changes in slope are common to the increasing temperature data of both

figures where the indium-gallium bond was used. One abrupt change in slope occurred between 200°C and 300°C and a second abrupt change occurred between 600°C and 800°C.

The changes occurring at 600°C and 800°C can be explained by referring to a phase diagram of the U - 2 Mo composition. From the phase diagram U - 2 Mo should be a two phase ($\alpha + \delta$) alloy until heated above 565°C. From 565°C to 639°C the intermetallic phases are $\alpha + \gamma$. The α , δ , and γ phases are respectively bcc, tetragonal, and orthorhombic. From 639°C to ~ 727°C, the $\beta + \gamma$ phases occur where β is a second tetragonal structure. Above ~ 727°C a single γ phase exists.

The ultrasonic data were not collected with small enough temperature increments to resolve the individual transitions to each one of these phases. However, the total effect of the phase transitions is reflected in the abrupt change in slope of the ultrasonic data from 600°C to 800°C. In the case of the shear wave, a very substantial change in wave velocity was observed at these phase transitions. There was also a very large increase in the attenuation of the shear wave from 650°C to 742°C which was the highest temperature at which a discernible through-transmission signal could be seen to make a measurement. The longitudinal wave also showed a marked increase in attenuation beginning just before the measurement at 780°C; but, the longitudinal wave amplitude at 800°C was sufficient that measurements to higher temperatures may have been possible. On decreasing temperature, the ultrasonic data showed the same abrupt change in slope from 800°C to 550°C where a slight hysteresis in the data appeared because of too rapid a change in temperatures between readings.

Finally, the abrupt change in slope from 200°C to 300°C observed on increasing temperature did not repeat itself for either the longitudinal data

or the shear data. Since no abrupt change in slope within this temperature regime is expected in U - 2 Mo, the influence of the indium-gallium bond on the data was suspect. Three individual tests were conducted on two separate specimens using the indium-gallium bond, a longitudinal test and a shear test on specimen 512, and a shear test on specimen 511. All three tests exhibited an abrupt change in data from 200°C to 300°C on increasing temperature only.

A second longitudinal test was conducted on specimen 512 where the bond between the specimen and the buffer rods consisted of a silicone based high temperature couplant HITEMPCO-1000⁷. As displayed in Figure 9, the normalized travel time versus temperature for this bond was linear from 25°C to 397°C with no abrupt changes in data from 200°C to 300°C. The slope of the data for this bond agrees very well with the initial slope of the longitudinal test where the indium-gallium bond was used. The final slope on decreasing temperature of the longitudinal test with the indium-gallium bond also agrees with the initial slope.

It is therefore concluded that the abrupt change in the ultrasonic data between 200°C and 300°C is a result of diffusion of the indium and gallium into the U - 2 Mo specimen and the Cupronickel 10 alloy buffer rods. Both indium and gallium form compounds UIn_3 and UGa_3 which are stable to 1300°C. The reaction is apparently completed at 300°C and a solid bond between the specimen and the buffer rods is in effect for the remainder of the test.

To overcome these experimental difficulties, the temperature dependence of the longitudinal velocity was calculated by using the normalized travel time data set of the HITEMPCO-1000 bond from 20°C to 397°C, the increasing temperature data set of the indium-gallium bond from 426°C to 802°C, and

the entire decreasing temperature data set from 802°C to 22°C. Each data set was normalized separately to its corresponding 25°C value. The shear velocity was calculated by using the two data sets of the normalized travel time data of the increasing temperature data set from 410°C to 742°C and the decreasing temperature data set from 742°C to 16°C. Thus the anomalous variation of the travel time data from 200°C to 300°C on increasing temperature for the indium-gallium bond may be ignored in the calculation of the ultrasonic longitudinal velocity, the shear velocity, and the elastic moduli versus temperature.

VI. Summary

The ultrasonic longitudinal and shear wave velocities in 17-4 PH stainless steel and Uranium - 2 Mo (weight percent) were determined from -40°C to 800°C. Young's modulus, shear modulus, and Poisson's ratio were calculated from the ultrasonic velocity data. Deviation of the ultrasonic data from monotonic dependence on temperature was explained with reference to phase changes in the materials. The indium-gallium bond between the specimen and buffer rods caused a significant offset in the ultrasonic travel time measurements of the U - 2 Mo tests on increasing temperatures from 200°C to 300°C.

FIGURE 1 - LONGITUDINAL AND SHEAR VELOCITY VERSUS TEMPERATURE FOR 17-4 PH STAINLESS STEEL

FIGURE 2 - YOUNG'S MODULUS VERSUS TEMPERATURE FOR
17-4 PH STAINLESS STEEL

FIGURE 3 - SHEAR MODULUS VERSUS TEMPERATURE FOR 17-4 PH STAINLESS STEEL

FIGURE 4 - POISSON'S RATIO VERSUS TEMPERATURE FOR 17-4 PH STAINLESS STEEL

FIGURE 5 - LONGITUDINAL AND SHEAR VELOCITY VERSUS TEMPERATURE
FOR URANIUM - 2 MOLYBDENUM

FIGURE 6 - YOUNG'S MODULUS VERSUS TEMPERATURE FOR
URANIUM - 2 MOLYBDENUM

FIGURE 7 - SHEAR MODULUS VERSUS TEMPERATURE FOR
URANIUM - 2 MOLYBDENUM

FIGURE 8 - POISSON'S RATIO VERSUS TEMPERATURE FOR
URANIUM - 2 MOLYBDENUM

FIGURE 9 - NORMALIZED LONGITUDINAL TRAVEL TIME VERSUS TEMPERATURE FOR URANIUM - 2 MOLYBDENUM

FIGURE 10 - NORMALIZED SHEAR TRAVEL TIME VERSUS TEMPERATURE
FOR URANIUM - 2 MOLYBDENUM

TABLE 1

ELEVATED TEMPERATURE DEPENDENCE OF THE ELASTIC PROPERTIES OF
17-4 PH STAINLESS STEEL IN THE ORDER DATA WAS RECORDED

17-4PH STAINLESS STEEL ULTRASONIC VELOCITY AND ELASTIC MODULI

DEGREE C	SHEAR VELOCITY MM/MSEC	SHEAR MODULUS MPSI	DEGREE C	LONG. VELOCITY MM/MSEC	YOUNG'S MODULUS MPSI	SHEAR MODULUS MPSI	POISSON'S RATIO
28	3.193	11.42	24	5.898	29.62	11.47	.291
109	3.145	11.07	228	5.768	27.78	10.72	.296
222	3.126	10.87	221	5.806	28.23	10.9	.295
309	3.068	10.43	309	5.876	27.81	10.44	.294
402	3.004	9.98	307	5.898	27.88	10.45	.296
520	2.911	9.34	377	5.72	26.43	10.12	.306
608	2.757	8.36	377	5.693	26.38	10.12	.304
612	2.766	8.41	469	5.599	25.24	9.68	.307
709	2.629	7.58	544	5.581	23.89	9.04	.321
755	2.567	7.22	612	5.395	22.23	8.41	.322
804	2.511	6.89	684	5.297	20.64	7.75	.332
861	2.679	7.89	758	5.273	19.44	7.23	.344
568	2.889	8.69	784	5.216	18.68	6.93	.348
594	2.984	9.3	878	5.123	17.11	6.3	.359
476	2.931	9.49	889	5.142	17.01	6.25	.361
423	2.965	9.72	525	5.523	24.34	9.3	.389
359	3	9.97	455	5.71	25.57	9.72	.316
295	3.024	10.15	28	5.851	29.41	11.42	.288
250	3.034	10.23	-43	5.934	30.61	11.83	.283
-42	3.263	11.92	25	5.892	29.61	11.47	.291
25	3.2	11.47					

TABLE 2

ELEVATED TEMPERATURE DEPENDENCE OF THE ELASTIC PROPERTIES OF
URANIUM - 2 MOLYBDENUM (WEIGHT PERCENT)

98% URANIUM 2% MOLYBDENUM ULTRASONIC VELOCITY AND ELASTIC MODULI

DEGREE C	SHEAR VELOCITY MM/MSEC	SHEAR MODULUS MPa	DEGREE C	LONG. VELOCITY MM/MSEC	YOUNG'S MODULUS MPa	SHEAR MODULUS MPa	POISSON'S RATIO
410	1.7	7.52	20	3.38	25.5	10.22	.248
455	1.683	7.33	110	3.315	24.45	9.75	.254
506	1.664	7.03	102	3.304	23.94	9.5	.26
549	1.637	6.88	205	3.28	23.34	9.25	.262
595	1.608	6.58	254	3.245	22.8	8.1	.258
643	1.528	5.87	298	3.233	22.48	8.92	.261
686	1.453	5.27	344	3.218	21.77	8.58	.269
694	1.381	4.15	397	3.185	20.94	8.2	.277
719	1.17	3.31	334	3.22	21.91	8.85	.267
742	1.135	3.08	428	3.119	20.29	8	.268
837	1.349	4.59	472	3.109	19.76	7.75	.275
885	1.381	4.84	510	3.08	18.28	7.85	.287
887	1.391	4.93	560	3.05	17.88	6.8	.3
887	1.42	5.15	610	3.037	16.79	6.4	.312
888	1.563	6.24	658	3.012	14.51	5.4	.344
838	1.608	6.83	704	2.944	10.81	3.9	.387
824	1.639	6.9	787	2.828	10.8	3.8	.385
805	1.67	7.18	749	2.787	8.54	3.85	.4
479	1.705	7.5	781	2.755	7.88	2.5	.418
433	1.749	7.93	802	2.751	6.27	2.2	.426
375	1.801	8.47	770	2.766	7.61	2.7	.41
345	1.811	8.59	703	2.784	9.94	3.6	.381
319	1.829	8.79	847	2.853	11.97	4.35	.378
299	1.841	8.83	599	3.056	13.25	4.83	.372
273	1.852	9.08	508	3.142	18.54	7.1	.306
257	1.851	9.08	376	3.212	21.35	8.38	.274
241	1.853	9.13	335	3.255	22.03	8.85	.274
200	1.858	9.2	312	3.229	22.24	8.8	.284
195	1.87	9.33	285	3.254	22.57	8.82	.285
174	1.878	9.43	278	3.248	22.7	9	.281
141	1.896	9.64	259	3.249	22.91	9.1	.259
118	1.903	9.74	297	3.232	22.87	9.17	.253
108	1.907	9.78	219	3.289	23.18	9.18	.262
10	1.947	10.23	196	3.241	23.25	9.28	.253
-49	1.903	10.75	173	3.255	23.53	9.4	.252
25	1.945	10.21	22	3.332	25.38	10.22	.241
			-42	3.414	26.6	10.7	.243
			25	3.385	25.5	10.21	.249

TABLE 3

COEFFICIENTS OF THE LEAST-SQUARES FIT TO THE MODULI VERSUS
TEMPERATURE DATA FOR 17-4 PH SS AND U - 2 MO

Temperature Range -40°C to 800°C

Modulus Value in PSI x 10⁶

$$M = M_0 + AT + BT^2 + CT^3 + DT^4$$

	<u>M₀</u>	<u>A</u>	<u>B</u>	<u>C</u>	<u>D</u>
17-4 PH SS					
Young's Modulus	29.9070	-1.4283 x 10 ⁻²	.4370 x 10 ⁻⁴	- .1020 x 10 ⁻⁶	5.9194 x 10 ⁻¹¹
Shear Modulus	11.6142	- .7428 x 10 ⁻²	.2255 x 10 ⁻⁴	- .0479 x 10 ⁻⁶	2.7638 x 10 ⁻¹¹
Poisson's Ratio	.2878	5.2889 x 10 ⁻⁵	-1.6634 x 10 ⁻⁷	4.3418 x 10 ⁻¹⁰	-2.3652 x 10 ⁻¹³
U - 2 Mo					
Young's Modulus	25.8072	-2.0632 x 10 ⁻²	.7108 x 10 ⁻⁴	- .1531 x 10 ⁻⁶	7.2403 x 10 ⁻¹¹
Shear Modulus	10.3616	- .6750 x 10 ⁻²	.1680 x 10 ⁻⁴	- .0441 x 10 ⁻⁶	2.1759 x 10 ⁻¹¹
Poisson's Ratio	.2458	.0167 x 10 ⁻²	-9.5689 x 10 ⁻⁷	2.2961 x 10 ⁻⁹	-1.260 x 10 ⁻¹²

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