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SOME EFFECTS OF COLD-WORKING,
THERMAL AGING, AND REACTOR IRRADIATION
ON ULTRASONIC WAVES VELOCITY
AND ATTENUATION IN SOME AUSTENITIC STEEL
TENSILE-CREEP SPECIMENS

D. O. Hunter

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IN SOME AUSTENITIC STEEL TENSILE-CREEP SPECIMENS

By

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~~ABSTRACT~~

For nondestructive testing purposes, austenitic steel specimens which had been cold-worked, thermally aged or fast flux irradiated were measured for effects on ultrasonic waves velocity and attenuation. This report presents a summary of ultrasonic measurements made on tensile-creep specimens of irradiated Types 304 and 348 stainless steel (SS), cold-worked Types 304 and 316 SS and thermally aged Type 304 SS. Cold-working was found to affect both attenuation and velocity; the direction and amount of change depends on the type of steel alloy, the percentage of cold-working and the frequency of ultrasonic waves. Thermal aging had negligible effect on velocity and attenuation. Neutron bombardment to high fluences (0.85×10^{22} to 3.7×10^{22} nvt) at high irradiation temperatures (825 to 1180 °F) typically causes decreases in velocity.

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D. O. Hunter

INTRODUCTION

Miniature buttonhead tensile specimens have sometimes been used at Battelle-Northwest for studies of various mechanical properties. Some of the austenitic steel specimens have become available for measurements of changes in ultrasonic velocity and attenuation as functions of the parameter of interest, i.e., neutron irradiation, thermal aging and cold-working. Knowledge of the effects of these manufacturing and environmental parameters on ultrasonic waves propagation is expected to be ultimately useful for nondestructive testing purposes, particularly for those that pertain to metals used in high temperature nuclear reactor environments.

This report includes a summary of ultrasonic measurements made on irradiated Types 304 and 348 SS specimens, cold-worked Types 304 and 316 SS specimens, and thermally aged Type 304 SS specimens.

SUMMARY AND CONCLUSIONS

The combined effects of neutron irradiation from 0.85×10^{22} to 3.7×10^{22} nvt (at all energies) and irradiation temperature from 825 to 1180 °F typically cause a decrease in ultrasonic wave velocity. Greatest decreases were observed for the highest fluence and highest temperature Type 348 SS specimens; decreases were less and more scattered for the Type 304 SS specimens.

The effects of thermal aging 304 SS specimens for 3045 hr at temperatures of 800, 900, 1000, 1100, 1200, and 1400 °F on propagation velocity is negligible within the given range.

There may be a slight increase in velocity relative to 304 SS which has received no thermal aging.

The effects of cold-working (CW) 316 SS 5, 10, and 20% was to decrease velocity (in the direction of grains elongation) with the greatest decrease at 5% CW and the least at 20% CW. For 304 SS, velocity decreased for 5 and 10% CW, but increased for 20% CW. For both metals, attenuation increased for 5% CW, remained about the same for 10% CW, and generally decreased for 20% CW. Attenuation at 20% CW was quite frequency dependent, which indicates that changes in grain size and/or shape plays an important role in determining the effects of cold-working on ultrasonic waves propagation.

No measurements were taken on specimens prior to their processing (irradiation, etc.). Accordingly, changes are relative to reference specimens arbitrarily selected. Measurements on twenty-five 304 SS and twenty-five 316 SS annealed specimens (with no further processing) indicates error caused by variations in materials and dimensions will be significant. Good accuracies will not be attainable unless measurements are made prior to processing so that a firm reference base is available for measurements following the processing.

EXPERIMENTAL PROCEDURE

The length of each miniature tensile specimen (see Figure 1a) is measured with a micrometer. For the irradiated specimens, three to six separate length measurements were taken remotely, and the smallest measurement used for subsequent calculations. Length measurements are also made for the reference specimens.

The time for an ultrasonic pulse to travel from a transmit crystal to a receive crystal through a reference specimen is established (as shown in Figure 1b). The position of the pulse is established at a reference position on a delayed sweep

FIGURE 1. Miniature Buttonhead Tensile Specimen and Method of Measuring Ultrasonic Pulse Through-Transmission Propagation Time

oscilloscope; typically, a zero crossing point on the second or third cycle is used as the reference. The reference specimen is then replaced by the specimen of interest and the change in time to the reference point on the through-transmission waveform is measured. The procedure is repeated one or two more times, with the specimen rotated somewhat in the specimen holder, so that an average may be obtained.

The change in velocity is then calculated using the following expression:

$$\Delta V = \frac{d_2}{T_1 + \frac{d_2 - d_1}{V_w} + \Delta T} - V_1$$

where

$$\Delta V = V_2 - V_1$$

V_2 = velocity in specimen to be measured

V_1 = velocity in reference specimen

d_2 = length of specimen to be measured

d_1 = length of reference specimen

V_w = velocity in water ($\sim 0.0581 \times 10^6$ in./sec)

$$T_1 = \frac{d_1}{V_1} \quad \text{and} \quad T_2 = \frac{d_2}{V_2}$$

$\Delta T = T_B - T_A$ where

$$T_B = [(d_w - d_2)/V_w] + T_2 \quad \text{(combined propagation time through water and the specimen to be measured)}$$

$$T_A = [(d_w - d_1)/V_w] + T_1 \quad \text{(combined propagation time through water and the reference specimen)}$$

d_w = water path distance between crystals.

In addition to determining ΔV (expressed as a percentage, $\Delta V/V_1$) some measurements of through transmission signal amplitude were made.

Spectrum analysis of the first few cycles of the through-transmission signal were made on some of the specimens. This was accomplished by using an RF gate (positioned to pass only the first few cycles of the through-transmission signal) and then feeding the gated signal into a Hewlett Packard 8553L/8552A Spectrum Analyzer.

RESULTS

EFFECTS OF COLD-WORKING ON ULTRASONIC WAVES PROPAGATION VELOCITY, LONGITUDINAL DIRECTION

Cold-working was accomplished on both 304 and 316 SS specimens by swaging. Elongated grains direction is the axial direction on the specimens. Twenty-three to 26 specimens for each material were cold-worked 5%, a similar number were cold-worked 10%, 20%, and also left unworked (annealed).

Measurements were made three times using a different set of send-receive crystals each time. Center frequencies during the three sets of measurements were 2.2, 5.5, and 10 MHz. Tuned preamplifiers were used to peak the through-transmission signal.

Change-in-velocity ($\Delta V/V$) figures for each group of 304 and 316 SS specimens are shown in Table 1. Trends for both materials are for velocities to decrease for 5% CW, but then to turn in the increasing direction for 10% and 20% CW.

Histograms of $\Delta V/V$ for both materials are shown in Figures 2 through 7. The indicated mean velocities are not particularly meaningful as absolute quantities since dispersion and frequency dependent attenuation are both present.

TABLE 1. Change in Ultrasonic Waves Velocity in Cold-Worked 304 and 316 Stainless Steel

<u>Material and % CW</u>	$\Delta V/V, \%$ (a) <u>Measurement Frequency</u> (b)		
	<u>2.2 MHz</u>	<u>5.5 MHz</u>	<u>10 MHz</u>
304 SS, Annealed	0	0	0
304 SS, 5%	-0.37	-0.40	-0.39
304 SS, 10%	-0.42	-0.24	-0.37
304 SS, 20%	+0.22	+0.22	+0.25
316 SS Annealed	0	0	0
316 SS, 5%	-0.77	-0.53	-0.53
316 SS, 10%	-0.60	-0.33	-0.43
316 SS, 20%	-0.51	-0.18	-0.20

a. Listed number is the arithmetical mean of the $\Delta V/V$ measurements made along the longitudinal axis on all the specimens in a group (23 to 26 specimens in each group).

b. Center frequency in the pulsed send-receive system.

EFFECTS OF COLD-WORKING ON ULTRASONIC WAVES PROPAGATION VELOCITY, TRANSVERSE DIRECTION

Velocity measurements, in a direction perpendicular to the direction of elongated grains, were made on quantities of about eighteen each of 304 and 316 SS, annealed, 5%, 10%, and 20% CW specimens. Accuracy of the measurements are mediocre because of the short distance (≤ 0.250 in.) traveled in the metal, and also because of the small-radius curved surface of the buttonheads. The results shown in Figure 8 for both 304 and 316 SS represent the average of two measurements on each specimen, one on each buttonhead.

FIGURE 2. Velocity Distribution of 304 Stainless Steel Cold Worked Specimens - Measured at 2.2 MHz

FIGURE 3. Velocity Distribution of 304 Stainless Steel Cold Worked Specimens - Measured at 5.5 MHz

FIGURE 4. Velocity Distribution of 304 Stainless Steel Cold Worked Specimens - Measured at 10 MHz

FIGURE 5. Velocity Distribution of 316 Stainless Steel Cold Worked Specimens - Measured at 2.2 MHz

FIGURE 6. Velocity Distribution of 316 Stainless Steel Cold Worked Specimens - Measured at 5.5 MHz

FIGURE 7. Velocity Distribution of 316 Stainless Steel Cold Worked Specimens - Measured at 10 MHz

FIGURE 8. Change in Compressional Velocity - Measured in Transverse Direction

EFFECTS OF COLD-WORKING ON ULTRASONIC WAVES ATTENUATION, LONGITUDINAL DIRECTION

Cold-working to 5% causes a greater signal loss, relative to annealed material, for both 304 and 316 SS (see Figures 9 and 10). At 10% and 20% CW, the direction of attenuation change relative to annealed material becomes frequency and material dependent; at 5.5 and 10 MHz, attenuation decreases for both 304 and 316 SS. The greatest changes occur when measurements are made at 10 MHz:

EFFECTS OF THERMAL AGING ON ULTRASONIC WAVES PROPAGATION VELOCITY

Velocity measurements, relative to an annealed 304 SS reference specimen, (the same reference specimen that was used for cold-worked specimens measurements) were made on groups of annealed 304 SS miniature tensile specimens. Each group had been thermally aged for 3045 hr: one group at 800 °F, another group at 900 °F, at 1000 °F, at 1100 °F, at 1200 °F, and at 1400 °F. Prior to thermal aging, all specimens had been cold-worked 27%, then annealed at 1950 °F for 1 hr followed by air cooling. Quantities for each group varied from seven to eighteen. Attenuation data were not taken; no change-in-attenuation trends were observed, however, at the center frequency used for velocity measurements (~4.2 MHz).

Results are shown as histograms in Figure 11. Velocity changes are considered to be negligible. Any velocity variations are within experimental error, part of which is the normal velocity variations to be expected in a group of specimens with similar fabrication history. No measurements were made before thermal aging. Compared to the group of 304 SS annealed specimens used for cold-work measurements, mean velocities for all the thermally aged groups increased slightly--typically less than 0.1% compared to the mean velocity of annealed specimens measured at 5.5 MHz.

FIGURE 9. Effects of Cold Working on Ultrasonic Signal Amplitude for 304 Stainless Steel at Three Frequencies

FIGURE 10. Effects of Cold Working on Ultrasonic Signal Amplitude for 316 Stainless Steel at Three Frequencies

FIGURE 11. Velocity Distribution of 304 Stainless Steel Specimens Thermally Aged for 3045 Hours

EFFECTS OF REACTOR IRRADIATION ON ULTRASONIC WAVES PROPAGATION VELOCITY

The miniature tensile specimens used for these measurements were part of a larger group being irradiated in sodium at the EBR-II, Row 2, Subassembly X-018. Included in the group used for velocity measurements were twenty-five 304 SS and forty-six 348 SS specimens. Prior to irradiation, the specimens had been annealed at 1950 °F for 1 hr followed by air cooling. The specimens were mounted in a holder such that eleven groups of seven each were located at varying distances from the reactor's midplane. Neutron fluence for the two groups closest to the midplane was estimated at 3.7×10^{22} nvt (all energies), and neutron fluence for the group farthest from the midplane was estimated at 0.85×10^{22} nvt (all energies).⁽¹⁾ Fluence for the remaining specimens lay between 0.85×10^{22} nvt and 3.7×10^{22} nvt, depending on their position. Irradiation temperatures for the eleven groups varied from an estimated 1130 to 1190 °F at midplane, to 825 °F at the position farthest from the midplane. Additional metallurgical and mechanical testing data may be found in Reference 1.

Experimental error for velocity measurements is expected to be greater for the irradiated specimens than for the cold-worked and thermally aged specimens. The main reason for the greater error is that it was impossible to closely inspect the irradiated specimens for burrs or dents on the ends or for cocking in the micrometer jaws. A special holder was fabricated for holding alignment during micrometer measurements, and a spotting scope was used to detect cocking and to search for burrs. When a burr was found, the specimen was maneuvered in the micrometer jaws so as to avoid the burr. The specimens were ultrasonically cleaned just prior to measurement. Micrometer measurements were repeatedly made on each specimen (after rotating the specimen slightly in its holder) until some

reproducibility was in evidence. The smallest measurement of each specimen was used for the calculations of change in velocity. If an error was made in length measurement, the measured length was probably slightly longer than actual length. The effect of this error would be to make a change in velocity (ΔV) appear to be too far in the decreasing direction. In most instances, however, an error of greater than 0.001 in. is required to make a significant difference in the calculated ΔV . In trial runs on unirradiated specimens, measurement error seldom exceeded 0.0005 in. on specimens without burrs.

304 Stainless Steel

A different reference specimen was used for these specimens than was used for the cold-worked and thermally aged 304 SS specimens. The reference specimen differed in that it had no mounting holes on the buttonheads, but was similar to the other reference specimen in that it too was solution annealed. Ultrasonic velocity measurements were carried out at a center frequency of 4.2 MHz. Velocity in the reference specimen was 203,696 in./sec.

Results for 304 SS are shown in Table 2. Changes in velocity, $\Delta V/V$, are generally in the negative (decreasing) direction. There may be a tendency for a greater change as fluence (and irradiation temperature) increases. Scatter is high. Also shown in Table 2 are the specimens lengths and the direction of change of the ultrasonic through-transmission signal strength relative to that of the standard. Note that many specimens apparently are longer than the 1.885 in. maximum specified for unirradiated specimens.

348 Stainless Steel

Ultrasonic velocity measurements were carried out at a center frequency of 10 MHz. A higher frequency was possible' for 348 SS since these specimens had a much finer grain structure

TABLE 2. Change in Velocity in Irradiated 304 Stainless Steel

Specimen Number	Neutron Fluence n/cm ² (All Energies) (a)	Irradiation Temperature, °F (a)	Length, in.	$\Delta V/V$, %	Through Transmission Signal Amplitude (b)
5-1	0.85 × 10 ²²	825	1.8792	-0.26	D
5-2	0.85	825	1.8759	+0.73	D
6-1	1.0	825	1.8773	+0.37	I
6-2	1.0	825	1.8803	+0.19	D
6-3	1.0	825	1.8878	+0.06	I
6-4	1.0	825	1.8777	+0.21	I
7-1	1.2	825	1.8836	-0.91	D
8-1	1.4	845	1.8781	+0.30	D
8-2	1.4	845	1.8801	0	D
8-3	1.4	845	1.8784	-0.37	D
10-1	2.0	1010	1.8869	-0.15	I
10-2	2.0	1010	1.8786	+0.10	I
10-3	2.0	1010	1.8843	-0.02	D
10-4	2.0	1010	1.8852	-0.14	I
11-1	2.6	1070	1.8794	-0.18	I
12-1	3.2	1110	1.8826	-0.47	I
13-1	3.5	1125	1.8802	-0.28	I
13-2	3.5	1125	1.8813	-0.64	I
13-3	3.5	1125	1.8758	-0.08	D
13-4	3.5	1125	1.8744	-0.59	D
14-1	3.7	1190	1.8796	-0.21	I
14-2	3.7	1140	1.8752	-0.70	I
15-1	3.7	1140	1.8854	-0.33	I
15-2	3.7	1140	1.8896	-0.45	I
15-3	3.7	1140	1.8784	-0.58	I

a. Data furnished by A. L. Ward and A. J. Lovell,
Battelle-Northwest

b. D = decreased relative to unirradiated reference standard
I = increased relative to unirradiated reference standard

than did the 304 SS specimens. At 10 MHz, velocity in the reference standard was approximately 218,151 in./sec; again, a figure of no particular significance because of dispersion.

Results for 348 SS are shown in Table 3. Changes in velocity are generally in the negative direction. Scatter is very high, but changes appear to be somewhat greater for the higher fluence and irradiation temperature specimens. Also shown in Table 3 are the through-transmission signal attenuations relative to the standard and specimen lengths.

Velocities distributions of the twenty-five 304 SS specimens and forty-six 348 SS specimens are shown in Figure 12. Note that the scale has been changed relative to those for cold-worked and thermally aged specimens.

SPECTRAL CONTENT OF THROUGH TRANSMISSION SIGNALS

The first few cycles of the through-transmission waveform were gated through a linear RF gate and then fed to a spectrum analyzer. Repetitive pulses are necessary to obtain reasonable contrast on the display screen of the analyzer. A center frequency of about 4 to 4.5 MHz was selected for most of the measurements, partly because it was known that signal attenuation would rapidly increase for 304 SS at higher frequencies. A center frequency of 10 MHz was used on some of the irradiated 348 SS specimens. Wide-band amplifiers and pulser, and damped PZT-5 crystals were used.

Spectral Content, Cold Worked 304 Stainless Steel

Spectral content photographs for cold-worked 304 SS are seen in Figure 13. The upper part of each photograph shows the first 10 to 15 cycles of the through transmission waveform. The middle part of each photo shows the gated waveform which is fed into the spectrum analyzer. Center frequency is 3.0 MHz; at 0.5 MHz/division, $f_{\min} = 0.5 \text{ MHz}$ and $f_{\max} = 5.5 \text{ MHz}$.

TABLE 3. Change in Velocity in Irradiated 348 Stainless Steel

Specimen Number	Neutron Fluence n/cm ² (All Energies) (a)	Irradiation Temperature, °F (a)	Length, in.	$\Delta V/V$, %	Through Transmission Signal Amplitude (b)
5-1	0.85×10^{22}	825	1.8697	-0.04	I
5-2	0.85	825	1.8730	-0.75	I
5-3	0.85	825	1.8752	-0.39	I
5-4	0.85	825	1.8755	-0.63	I
5-5	0.85	825	1.8765	-0.16	D
6-1	1.0	825	1.8765	+0.38	I
6-2	1.0	825	1.8792	+0.08	I
6-3	1.0	825	1.8724	-0.69	I
7-1	1.2	825	1.8826	-0.23	I
7-2	1.2	825	1.8756	-0.62	I
7-3	1.2	825	1.8776	-1.40	I
7-4	1.2	825	1.8811	+0.37	D
7-5	1.2	825	1.8773	-0.57	I
7-6	1.2	825	1.8795	-0.79	I
8-1	1.4	845	1.8847	-0.08	I
8-2	1.4	845	1.8748	-0.78	I
8-3	1.4	845	1.8804	-0.99	I
9-1	1.6	930	1.8809	+0.17	I
9-2	1.6	930	1.8805	-0.23	I
9-3	1.6	930	1.8808	-0.25	I
10-1	2.0	1010	1.8794	-0.61	I
10-2	2.0	1010	1.8825	-0.25	I
10-3	2.0	1010	1.8818	-0.02	I
11-1	2.6	1070	1.8756	+0.91	I
11-2	2.6	1070	1.8901	-1.87	I
11-3	2.6	1070	1.8778	-0.13	I
11-4	2.6	1070	1.8860	-1.76	I
11-5	2.6	1070	1.8819	-1.32	I
11-6	2.6	1070	1.8764	-0.68	I
12-1	3.2	1110	1.8771	-1.04	I
12-2	3.2	1110	1.8781	-0.67	I
12-3	3.2	1110	1.8797	-0.52	I

TABLE 3. (contd)

Specimen Number	Neutron Fluence n/cm ² (All Energies) (a)	Irradiation Temperature, °F(a)	Length, in.	$\Delta V/V$, %	Through Transmission Signal Amplitude(b)
12-4	3.2×10^{22}	1110	1.8840	-0.12	I
12-5	3.2	1110	1.8804	-1.79	I
12-6	3.2	1110	1.8849	-0.90	I
13-1	3.5	1125	1.8853	-0.90	D
13-2	3.5	1125	1.8797	-0.24	I
13-3	3.5	1125	1.8886	-0.25	D
14-1	3.5	1140	1.8933	-1.79	I
14-2	3.5	1140	1.8851	-0.91	I
14-3	3.5	1140	1.8915	-0.96	D
14-4	3.5	1140	1.8897	-0.97	I
14-5	3.5	1140	1.8834	-1.75	I
15-1	3.5	1140	1.8895	-2.41	I
15-2	3.5	1140	1.8876	-1.44	I
15-3	3.5	1140	1.8899	-1.39	I

a. Data furnished by A. L. Ward and A. J. Lovell,
Battelle-Northwest

b. D = decreased relative to unirradiated reference standard
I = increased relative to unirradiated reference standard

Typical photographs are shown for annealed, 5%, 10%, and 20% CW specimens. Changes in attenuation do not appear to be strongly frequency dependent in the 0.5 to 5.5 MHz range; although, as shown in Figure 9, the decrease in attenuation for 10% and 20% CW specimens, relative to annealed and 5% CW specimens, does not show up at the lower frequencies (i.e., 1.5 to 2.5 MHz).

Spectral Content, Thermally Aged 304 Stainless Steel

The same pulser, amplifiers, and piezoelectric crystals as were used for the cold-worked specimens. Results are shown in Figure 14. No significant differences are noted as a function of aging temperature.

FIGURE 12. Velocity distribution of 304 and 348 Stainless Steels Specimens Irradiated at EPR-II, 0.85-37 x 10²² nwt (TOTAL), 82F to 1190 °F

FIGURE 13. Ultrasonic Through-Transmission Signal Spectral Content, 304 Stainless Steel Cold-Worked

FIGURE 14. Ultrasonic Through-Transmission Signal Spectral Content, 304 Stainless Steel Thermally Aged for 3045 Hours

Spectral Content, Irradiated 304 Stainless Steel

The same pulser, amplifiers, and crystals were used as were used for the cold-worked specimens. Results are seen in Figure 15. The irradiated specimens did not have holes on the ends (see Figure 1) as did the cold-worked and thermally aged specimens (this accounts for some basic differences in their respective spectral contents). The most notable difference in the spectral content of the irradiated specimens relative to their unirradiated reference is the much more rapid drop-off of the frequency components higher than about 5 MHz. No particular differences are noted between spectrums on specimens at the high and low ends of the neutron fluence range with one exception: the highest temperature 304 SS specimen (the center specimen at the No. 14 level in the reactor) had the greatest loss of high frequency components of any of the irradiated 304 SS specimens.

Spectral Content, Irradiated 348 Steel

A higher frequency (10 MHz) was selected for the 348 SS specimens since their finer grain structure permitted use of a higher frequency without excessive attenuation. Results are shown in Figure 16. It is not known that irradiation had an effect of decreasing attenuation as indicated by the photographs in Figure 16. Only one unirradiated reference specimen was available and it may have been nontypical. For these photographs, center frequency is 10 MHz, $f_{\min} = 7.5$ MHz, and $f_{\max} = 12.5$ MHz.

No significant trends are noted in these photographs. Two or three specimens (not shown in Figure 16) had their response peaks shifted 0.3 to 0.4 MHz in the increasing frequency direction.

FIGURE 15. Ultrasonic Through-Transmission Signal Spectral Content, 304 Stainless Steel Reactor Irradiated

- (1) NEUTRON FLUENCE, ALL ENERGIES
(2) IRRADIATION TEMPERATURE

FIGURE 14. Ultrasonic Through-Transmission Signal Spectral Content, 348 Stainless Steel Reactor Irradiated

DISCUSSION OF RESULTS

The objectives of this investigation have been limited primarily to a presentation of the results. An in-depth study must await better test conditions; i.e., control over all parameters which might affect the measurement of interest, and the use of specimens with favorable shape and dimensions. Such a study is now in progress for cold-worked 316 SS.⁽²⁾ In addition, the measurements of interest should be taken both before and after the processing under study. Without these test conditions, data will be scattered; thus, a comprehensive discussion is not justified. Discussion, then, will be short and generalized.

EFFECTS OF COLD-WORKING

An increase in dislocations density is often associated with a simultaneous increase in attenuation and a decrease in velocity. Such might be the case at 5% cold-working. As percentage of cold-working increases, at least two changes may influence ultrasonic waves. First, grain shapes become elongated in the longitudinal direction and smaller in the transverse direction. Second, microstructure defects have become so numerous as to effectively pin dislocations, thus resulting in a decrease in attenuation and an increase in velocity. The change of grain dimensions would be expected to affect high frequency measurements more so than low frequency measurements.

EFFECTS' OF THERMAL-AGING

Although the aging temperatures were too low to allow significant grain growth, some carbide precipitation and sigma phase formation may take place.⁽³⁾ An effect of carbide precipitation could be to increase the apparent elastic constants by restricting dislocations movements which in turn would increase ultrasonic waves velocity. Others have noticed a slight increase in Young's modulus in 304 SS steel for room temperature measurements after the steel had been heated to elevated temperatures.⁽⁴⁾

EFFECTS OF REACTOR IRRADIATION

Other ultrasonic measurements have been made on irradiated 304 SS, both after high and low irradiation temperatures,⁽⁵⁾ but no measurements have yet been reported on the same specimens both before and after irradiation. The very large amount of scatter (even among specimens located alongside each other in the reactor) certainly complicates any analysis. It has been previously noted that irradiation temperature apparently plays a more important role than does neutron fluence in affecting elastic properties in a low alloy steel.⁽¹⁾ Perhaps some of the scatter was caused by unsuspected temperature variations due to differences in sodium flow past the tightly packed specimens. In any event, decreases in velocity seem to predominate--in agreement with earlier measurements.⁽⁵⁾ The decrease in velocity is apparently associated with the presence of displacement damage (i.e., dislocation loops and voids).

Another form of damage, weakening of grain boundaries due to collection of helium atoms, is potentially more dangerous than displacement type damage since the helium agglomeration occurs primarily at higher temperatures (operating temperatures of fast breeder reactors). The spectrum analysis taken on the irradiated specimens was intended to test a theory that weakened grain boundaries would be more apt to show up at a test frequency where half period is about equal to mean travel time per grain; the weakened grain would, perhaps, consume more energy in vibrating much as would an unpinned dislocation line. For No. 4 grain size 304 SS this frequency would be in the region of 2 to 5 MHz, and for No. 8 grain size 348 SS, the corresponding frequency would be in the range of 8 to 20 MHz. Referring to Figure 15, for 304 SS there is some drop-off of response at 5 to 5.5 MHz for all specimens with the greatest decrease by the specimen with highest fluence and irradiation

temperature. A similar drop-off is not seen for 348 SS (Figure 16) but perhaps the reason is that the test frequency was too low ($f_0 = 10$ MHz).

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REFERENCES

1. Quarterly Progress Report, October, November, December, 1969. Reactor Fuels and Materials Development Programs, BNWL-1279. Battelle-Northwest, Richland, Washington, March, 1970.
2. F. L. Becker, R. L. Trantow. "Nondestructive Evaluation of Cladding Cold Work and Grain Size," Transactions of American Nuclear Society, Vol. 12-2. December, 1969.
3. Paul G. Nelson. "Constitution and Heat Treatment of Stainless Steels." An ASM Monograph, 1961.
4. Charles W. Andrews. "Effect of Temperature on the Modulus of Elasticity," Metals Progress, p. 85, July 1950.
5. D. O. Hunter. Ultrasonic Velocities and Critical Angle Method Changes in Irradiated 304 Stainless Steel, BNWL-1012. Battelle-Northwest, Richland, Washington. March, 1969.

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