**ULTRASONIC VELOCITIES
AND
CRITICAL-ANGLE-METHOD CHANGES
IN IRRADIATED 304 STAINLESS STEEL**

MARCH 1969

**AEC RESEARCH &
DEVELOPMENT REPORT**

FIGURE NO.	SCALE	DATE	REVISIONS

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3 3679 00061 2699

BNWL-1012

UC-40, Radiation
Effects on Materials

ULTRASONIC VELOCITIES AND CRITICAL-ANGLE-METHOD
CHANGES IN IRRADIATED 304 STAINLESS STEEL

By

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March 1969

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Printed in the United States of America

Available from

Clearinghouse for Federal Scientific and Technical Information
National Bureau of Standards, U. S. Department of Commerce
Springfield, Virginia 22151

Price: Printed Copy \$3.00; Microfiche \$0.65

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CHANGES IN IRRADIATED 304 STAINLESS STEEL

ABSTRACT

Ultrasonic critical-angle-method amplitude changes and ultrasonic shear and longitudinal velocities changes are measured on reactor irradiated AISI 304 stainless steel.

For specimens irradiated at temperatures between 140 °F and 350 °F, shear velocity decreased from about 0.1% at 1.5×10^{18} nvt > 1 MeV to about 1.25% at 1×10^{20} nvt > 1 MeV; longitudinal velocity decreased from about 0.1% to 0.5% for the same neutron fluences. Measurements of velocities changes for specimens irradiated at temperatures between 610 °F and 680 °F were inconclusive due to the small changes. There were indications, however, that high temperature irradiated specimens had experienced increased velocities before decreasing velocities trends were established.

The trends of critical-angle-method amplitude changes, as functions of irradiation parameters, are generally comparable to the trends of velocities changes.

Apparent changes in elastic constants are calculated by using measured changes in density and in shear and longitudinal velocities.

Measurement methods and apparatus are described.

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ULTRASONIC VELOCITIES AND CRITICAL-ANGLE-METHOD
CHANGES IN IRRADIATED 304 STAINLESS STEEL

D. O. Hunter

INTRODUCTION

Austenitic stainless steels have been shown to suffer loss of ductility when irradiated.^(1,2) Decrease in ductility is particularly severe, due to the combined effects of irradiation and temperature, at elevated temperatures although the greatest change between the irradiated and unirradiated conditions occurs at lower test temperatures, as shown in Figure 1. Degraded ductility in reactor structural steels may impose costly operational limitations and, if undetected, could lead to even more costly mechanical failures. A nondestructive method for measuring the irradiation effects leading to decreases in ductility would help alleviate the problems brought on by an unknown decrease in ductility.

This report includes results and a description of methods used to make ultrasonic measurements in neutron irradiated 304 stainless steel. Changes in shear and longitudinal velocities and critical-angle-method signal amplitude are determined as a function of neutron fluence >1 MeV, with irradiation temperature as a parameter.

SUMMARY

Measurements of changes in ultrasonic shear velocity, longitudinal velocity, and critical-angle-method signal amplitude were made on

FIGURE 1
The Effect of Test Temperature on the Ductility of Irradiated
AISI 304 Stainless Steel in the Annealed Condition

irradiated 304 steel. For specimens irradiated at temperatures between 140 °F and 350 °F:

- shear velocity decreased as a function of neutron fluence >1 MeV; $\Delta V_S/V_S$ varied from about -0.15% at 1.5×10^{18} nvt to about -4.25% at 1.0×10^{20} nvt;
- longitudinal velocity decreased as a function of neutron fluence >1 MeV; $\Delta V_L/V_L$ varied from about -0.12% at 1.5×10^{18} nvt to about -0.51% at 1.0×10^{20} nvt;
- critical-angle-method signal amplitude changed approximately in proportion to the velocity changes.

Decreases in velocities and density suggest decreases in shear, bulk, and Young's elastic moduli, and an increase in Poisson's ratio.

For specimens irradiated at temperatures between 610 °F and 680 °F, ultrasonic changes were too small to be conclusive. Velocity measurements indicated decreasing trends but with possible net increases at the lower neutron fluences.

CONCLUSIONS

The ultrasonic methods described herein are capable of detecting a type of irradiation damage which remains relatively stable at irradiation temperatures less than about 350 °F. Other forms of irradiation damage still stable at irradiation temperatures between 610 °F and 680 °F are not easily detected. Greater sensitivity to high temperature irradiation damage would result if reference measurements taken prior to irradiation were available during

post-irradiation measurements.

Besides applications related to measuring radiation damage to structural components or surveillance specimens, ultrasonic methods appear to be useful in the laboratory for studies pertaining to temperature related irradiation damage and damage recovery.

EXPERIMENTAL PROCEDURE

The irradiations and measurements to be described herein were conducted concurrently with irradiations and measurements on A302-B and A542-B low alloy steels. A detailed report pertaining to A302-B and A542-B steels has been separately issued.⁽³⁾

Specimens Preparation and Irradiation

Specimens were machined from donor plate No. B-55697, a $\frac{1}{2}$ in. thick plate included in the stockpile used for the "Irradiation Effects on Reactor Structural Materials Program". Fabrication history, briefly, is as follows:

- (1) hot rolled in 17 passes from 6 in. to $\frac{1}{2}$ in. plate at 2250 °F (start of rolling) to 1700 °F (end of rolling);
- (2) heated one hour at 2000 °F, then water quenched;
- (3) pickled, then abrasive cut to size.

Specimens were machined from the donor plate as shown in Figure 2. Before machining, the plate section was solution annealed at 1950 °F for one hour, in vacuum, and furnace cooled. After machining to the finished specimen, all specimens except one unirradiated control specimen were stress-relieved, 650 °F for

FIGURE 2
304 Test Specimens' Positions in Donor Plate

20 hours with furnace cooling. Typical Rockwell A hardness measurements following stress relieving were $R_A = 44$ to 46.

Finished specimens were encapsulated in 304 SS capsules as shown in Figure 3. A helium gap was used to enable control of specimen temperature by slowing transfer of gamma heat. In some specimen capsules, spacer wires were used, as shown in Figure 4, to assure uniform gap spacing. The individual encapsulated specimens were mounted in irradiation-capsules, as shown in Figures 5 and 6, for reactor irradiation.

Prior to finish machining, all specimens were identified with nomenclature as shown in Figure 2 and explained as follows:

- (1) The first character on the first line, a number, identifies material; "3" designates 304 steel ("1" designates A302-B, and "2" designates A542-B);
- (2) the second character on the first line, a letter, identifies irradiation temperature; "C" designates cold or 140 °F to 350 °F; "H" designates hot or 610 °F to 680 °F; all other letters pertain to unirradiated control specimens;
- (3) the third character on the first line, a number, identifies sequential position with respect to irradiation dosage; i.e., "1" received the least exposure and "7" received the most exposure;
- (4) the first character on the second line, a letter, designates the predominant rolling direction associated with the specimens'

FIGURE 3
Test Specimens in Various Stages of Encapsulation

FIGURE 4
Test Specimens Being Encapsulated with Wire Spacers

FIGURE 5
Typical Capsules used for ETR Reactor Ambient Temperature
Positions' Irradiations

FIGURE 6
Typical Quadrant Capsule Assemblies used for ETR Reactor
G7-High-Temperature-Loop Irradiations

long dimension (2.577 in.); "L" designates longitudinal rolling direction; transverse machined specimens ("T") were not irradiated;

- (5) the second character on the second line, a letter, pertains to surface position in donor plate and may be ignored for 304 since a specimen's cross-section dimension is about the same as donor plate thickness;
- (6) the third character on the second line, a number, pertains to control specimens identification (see Table VIII).

Positions of ultrasonic measurements on a test specimen are identified by referring to Figure 7. Specimens' sides and positions are noted in tables of data to follow and may be identified by referring to Figure 7.

Irradiation was done at the Experimental Test Reactor (ETR), National Reactor Test Station (NRTS), Idaho. Irradiation data is shown in Table I which includes data on low alloy steels as well. Deencapsulation and other post-irradiation handling was done at Pacific Northwest Laboratory (PNL). Determination of neutron exposure at each capsule reactor position was done by activation analysis of neutron monitors attached to each capsule.

Irradiation experiments were designed in such a manner that specimens were irradiated in one of two different temperature ranges: 140 °F to 350 °F and 610 °F to 680 °F. Temperature monitors were included in a dummy specimen in each capsule assembly so that

FIGURE 7
Example of Sides and Positions Locations, Both Irradiated and Unirradiated Specimens

TABLE I

NEUTRON IRRADIATION DATA ON TEST SPECIMENS

Test Speci- mens Series	Indic- ated Fast Flux Fe Wire, nv $\times 10^{14}$	Indic- ated Fast Flux Ni Wire, nv $\times 10^{14}$	Ave. of cor- rected Fast Flux, nv $\times 10^{14}$	Indic- ated Thermal Flux Al-Co Wire, nv $\times 10^{14}$	Cor- rected Thermal Flux, nv $\times 10^{14}$	Eff. Irrad. Time, Sec, $\times 10^6$	Fast Fluence >1 MeV, nvt ¹⁸ $\times 10^{18}$	Thermal Fluence 2200 m/sec., nvt ¹⁸ $\times 10^{18}$	Thermal to Fast Flux Ratio
A11 C1	0.0070	0.0080	0.0081	0.102	0.098	1.82	1.47	17.8	12.1:1
A11 C2	0.0137	0.0148	0.0153	0.153	0.145	1.82	2.78	26.4	9.5:1
A11 C3	0.0361	0.0378	0.0373	0.633	0.645	1.82	6.79	117.3	17.3:1
A11 C4*	0.0345	0.0362	0.0354	0.569	0.580	5.35	18.9	310.0	16.4:1
A11 C5	0.2220	0.2490	0.2850	0.934	0.882	1.79	51.0	158.0	3.1:1
A11 C6	0.5280	0.5200	0.5700	1.68	1.62	1.79	102.0	290.0	2.8:1
1LT, 1LM C7 & H7	1.080	0.9790	1.049	2.81	2.82	1.79	188.0	505.0	2.7:1
2LT, 3LT C6 & H7	1.060	0.9500	0.9800	3.07	3.13	1.79	175.0	560.0	3.2:1
1LT, 1LM H4	0.0355	0.0411	0.0454	0.138	0.119	4.19	19.0	49.9	2.6:1
2LT, 3LT H4	0.0352	0.0446	0.0472	0.139	0.120	4.19	19.8	50.3	2.5:1
1LT, 1LM H5	0.2380	0.2890	0.3120	0.552	0.475	5.34	166.0	254.0	1.5:1
2LT, 3LT H5	0.1760	0.1960	0.2210	0.490	0.421	5.34	118.0	224.0	1.9:1

* Estimated

irradiation temperatures could be approximately determined. A listing of indicated irradiation temperatures from post-irradiation examination of temperature monitors is shown in Table II for low alloy steels as well as 304 steel. Also listed in Table II are calculated irradiation temperatures for both the surface and the interior of each specimen.

Measurement Methods

Changes in Shear Velocity, $\Delta V_S/V_S$

Changes in shear velocity were measured as shown in Figure 8. A pulse incident in water on a specimen's surface at about 19° is mode converted to a shear wave at 45° . With a square cross-section, the pulse travels about the inside as shown. With each revolution, a portion of the pulse's energy leaves the specimen and travels to a receive crystal thus forming on the oscilloscope a series of pulses which are separated in time from each other by the travel time within the specimen. By referencing to one of these echoes on an unirradiated specimen, such as the fifth echo, a measure of change in shear velocity can be obtained by knowing the time position of the fifth echo on an irradiated specimen. After corrections are made for dimensional differences relative to the reference specimen, ΔV_S can be determined from the change in time position, ΔT_S .

Measurement frequency was about 3 MHz.

Changes in Longitudinal Velocity, $\Delta V_L/V_L$

Changes in longitudinal velocity were measured as shown in Figure 9.

TABLE II
INDICATED AND CALCULATED IRRADIATION
TEMPERATURE DATA ON TEST SPECIMENS

Test Speci- mens Series	Width of Helium Gap in.	Fast Flux nv $\times 10^{14}$	Estim. Gamma Heat watt/gm.	Coolant Water Temp. °F	Estim. Inter. Temp. (Calcu- lated) °F	Estim. Surface Temp. (Calcu- lated) °F	Indicated Surface Temp., T, (Temp. Monitors) °F
C1	0.0015	0.0081	0.116	140	142	141	T<312
C2	0.0015	0.0153	0.224	140	144	143	T<312
C3	0.0015	0.0373	0.535	140	149	145	T<312
C4	0.0025	0.0354	0.506	140	150	147	T<312
C5	0.0055	0.2850	4.08	140	270	242	T<312
C6	0.0015	0.5700	8.15	140	277	224	312<T<349
C7, 1LT & 1LM	0.0015	1.049	15.0	140	392	294	312<T<349
C7, 2LT & 3LT	0.0015	0.980	14.0	140	376	285	312<T<349
H7, 1LT & 1LM	0.0105	1.049	15.0	140	837	739	610<T<680
H7, 2LT & 3LT	0.0105	0.980	14.0	140	791	700	610<T<680
H4, 1LT & 1LM	0.0015	0.0454	0.650	550	561	557	610<T<680
H4, 2LT & 3LT	0.0015	0.0472	0.675	550	561	557	610<T<680
H5, 1LT & 1LM	0.0015	0.312	4.46	550	625	596	610<T<680
H5, 2LT & 3LT	0.0015	0.221	3.16	550	603	582	610<T<680

FIGURE 8
Method for Measuring Shear Velocity Changes in Irradiated Steel

FIGURE 9
Method for Measuring Longitudinal Velocity Changes in Irradiated Steel

A pulse enters the specimen at normal incidence and reverberates between two opposing faces. Due to the small size of the specimen, interference echoes rapidly build up preventing intelligible interpretation after four or five reverberations. Measurement of ΔT_L is made as on the shear velocity method; and ΔV_L is calculated after dimensional corrections are made.

Measurement frequency was about 3 MHz.

A similar method was used in some measurements on tensile specimens except that change in through-transmission time was measured.

Changes in Critical-Angle-Method Signal Amplitude, ΔA_{CA}

Changes in critical-angle-method signal amplitude were measured as shown in Figure 10. A transmit pulse of 20 to 40 oscillations at about 3 MHz is incident in water on the specimen's surface at the critical angle for surface wave generation — about 31° . At this angle, reflections of beam components vary rapidly in phase angle, relative to the incident beam, so that with nominal transmit-beam spread, phase angle changes approaching 360° become possible.⁽⁴⁾ In addition, a narrow portion of the beam is laterally displaced in the direction of propagation.⁽⁴⁾ By careful positioning of a receive transducer, it is possible to obtain almost complete cancellation of received beam components, in the central region of the pulse, due to the large changes in phase angle.⁽⁵⁾ Since the critical angle for surface waves is dependent upon density and

FIGURE 10
Critical-Angle Method for Measuring Ultrasonic Properties Changes in Irradiated Steel

elastic properties of a material, any slight change in these physical properties will cause a redistribution of reflected and re-radiated energy.⁽⁴⁾ The result is that cancellation of received beam components no longer occurs, and a signal appears whose amplitude is related to the changes in elastic properties.

In contrast to the shear and longitudinal velocities changes, which are affected by property changes through most of the specimen's thickness, critical angle changes are caused solely by changes in surface and near-surface properties.

Measurements were taken using two methods:

- (1) by measuring RF signal amplitude at a stationary position on the specimen, and
- (2) by integrating the RF signal amplitude over a scanned region of about one inch along the 2.577 in. dimension.

Averaging of data from these two methods gave the final data point. More weight was given the integrated scans since error from material scatter would tend to balance-out from results of this method.

Mechanical Apparatus

Measurements were made in the 324 Building Shielded Facility at PNL. A view of some of the handling apparatus is seen in Figure 11. Irradiated specimens are remotely removed from storage, ultrasonically cleaned, and loaded in the immersion tank shown at the right. After measurements are completed, specimens are stored in the cask shown in Figure 12.

FIGURE 11
Hot-Cell Specimen Handling Area Seen Through Hot-Cell Window

FIGURE 12
Gatling-Gun Specimens-Storage-Cask

RESULTS

Changes in Shear Velocity

A tabulation of measurements is shown in Table III. A final data point for a specimen is the scatter corrected average of measurements at three positions (positions 1-4, 2-5, and 3-6) as shown in Figure 7. A description of scatter appears subsequently. Final data points are plotted as curves in Figure 13.

Scatter and error shown in Figures 13, 14, and 16 refer to an estimated possible deviation of irradiated specimen's data points relative to the points plotted in the curves; or it may also show the estimated limits of the zero reference (unirradiated specimen).

From the curves in Figure 13, the following observations are noted:

- Neutron irradiation has caused a decrease in shear velocity in the approximate amount of 0.5% per decade in the range of 10^{18} nvt to 2×10^{20} nvt (>1 MeV) for low temperature reactor irradiations ($T \cong 140^\circ\text{F}$ to 350°F). Ignoring anomalies, the relationship may be expressed as follows:

$$\Delta V_S/V_S (\%) \cong -(A_S + 0.5 \log \frac{\phi_t}{10^{18}})$$

where $A_S \cong 0.1\%$, and ϕ_t = neutron fluence >1 MeV.

- Change in shear velocity appears to be in the decreasing direction for high temperature reactor irradiations ($T \cong 610^\circ\text{F}$ to 680°F). The position of the high temperature curve suggests the possibility that V_S had increased at lower fluences but

TABLE III

CHANGE IN SHEAR VELOCITY FOR IRRADIATED 304

Specimen Identi- fication	$\Delta V_S/V_S$			$\Delta V_S/V_S$	$\Delta V_S/V_S$	$\Delta V_S/V_S$	Average
	Position 1-4 %	Position 2-5 %	Position 3-6 %	Position 1-4 Scatter Cor- rected, %	Position 2-5 Scatter Cor- rected, %	Position 3-6 Scatter Cor- rected, %	Position All Pos- itions, Scatter Cor- rected, %
3C1LT	-0.114	-0.088	-0.003	-0.107	-0.147	-0.143	-0.132
3C2LT	-0.161	-0.183	-0.130	-0.193	-0.245	-0.241	-0.226
3C3LT	-0.515	-0.495	-0.353	-0.591	-0.547	-0.473	-0.537
3C4LT	-0.839	-0.837	-0.715	-0.904	-0.880	-0.865	-0.883
3C5LT	-0.986	-0.979	-1.040	-1.019	-1.022	-1.221	-1.087
3C6LT	-1.187	-1.174	-1.050	-1.330	-1.243	-1.206	-1.260
3C7LT	-0.856	-0.817	-0.847	-1.021	-0.907	-0.960	-0.963
3H4LT	+0.331	+0.266	+0.331	+0.282	+0.223	+0.189	+0.231
3H5LT	+0.104	+0.016	+0.163	+0.087	-0.027	-0.027	+0.011
3H7LT	+0.045	+0.177	-0.105	-0.055	+0.122	-0.183	-0.039

FIGURE 13
Change in Ultrasonic Shear Velocity in Irradiated 304

subsequently reversed direction at some fluence still lower than final dosages used in this study. Magnitude of changes are too small to be reliably isolated from possible scatter and error.

- If a sufficient number of measurements can be made so as to reduce scatter error, the radiation damage on low temperature irradiated 304 can be detected at exposures as low as 1 to 2×10^{18} nvt.
- The low temperature irradiated specimen at 1.75×10^{20} nvt has a velocity change much less than would be expected from the curve's trend.

Changes in Longitudinal Velocity

A tabulation of measurements is shown in Table IV. A final data point for a specimen is the scatter corrected average of measurements at six positions (positions 1 through 6) as shown in Figure 7. Final data points are plotted in Figure 14.

From the curves in Figure 14, the following observations are noted:

- Neutron irradiation has caused a decrease in longitudinal velocity in the approximate amount of 0.22% per decade in the range of 10^{18} nvt to 2×10^{20} nvt (>1 MeV) for low temperature reactor irradiations ($T \cong 140$ °F to 350 °F). Ignoring anomalies, the relationship may be expressed as follows:

$$\Delta V_L / V_L (\%) \cong -(A_L + 0.22 \log \frac{\phi t}{10^{18}})$$

where $A_L \cong 0.10\%$, and ϕt = neutron fluence >1 MeV.

TABLE-IV
CHANGE IN LONGITUDINAL VELOCITY
FOR IRRADIATED 304

Specimen Identi- fication	Change In Longitudinal Velocity, $\Delta V_L/V_L^*$						Average $\Delta V_L/V_L$ All Pos. Scatter Corrected - $(+0.033)$, %
	Posit- ion 1, %	Posit- ion 2, %	Posit- ion 3, %	Posit- ion 4, %	Posit- ion 5, %	Posit- ion 6, %	
3C1LT	-0.113	-0.047	-0.036	-0.123	-0.076	-0.121	-0.119
3C2LT	-0.206	-0.047	-0.127	-0.158	-0.148	-0.121	-0.168
3C3LT	-0.286	-0.253	-0.166	-0.300	-0.287	-0.254	-0.291
3C4LT	-0.406	-0.318	-0.279	-0.474	-0.445	-0.435	-0.426
3C5LT	-0.554	-0.301	-0.488	-0.503	-0.410	-0.507	-0.493
3C6LT	-0.590	-0.420	-0.405	-0.547	-0.495	-0.419	-0.512
3C7LT	-0.463	-0.254	-0.427	-0.311	-0.297	-0.259	-0.368
3H4LT	+0.094	+0.308	+0.163	+0.084	+0.065	+0.130	+0.108
3H5LT	+0.036	+0.098	+0.072	+0.058	+0.008	+0.108	+0.030
3H7LT	+0.022	+0.058	+0.029	+0.022	+0.058	+0.047	+0.006

* Measurements taken at six positions; see Figure-7

Neutron Fluence $nvt > 1 \text{ MeV}$

FIGURE 14

Change in Ultrasonic Longitudinal Velocity in Irradiated 304

- Change in longitudinal velocity appears to be in the decreasing direction for high temperature reactor irradiations ($T \cong 610^{\circ}\text{F}$ to 680°F). The position of the high temperature curve suggests the possibility that V_L had increased at lower fluences but subsequently reversed direction at some fluence still lower than final dosages used in this study. Magnitude of changes are too small to be reliably isolated from possible scatter and error.
- If a sufficient number of measurements can be made so as to reduce scatter error, the radiation damage on low temperature irradiated 304 can be detected at exposures as low as 4 or 5 $\times 10^{18}$ nvt > 1 MeV.
- The low temperature irradiated specimen at 1.75×10^{20} nvt has a velocity change much less than would be expected from the curve's trend.

Additional change-in-longitudinal-velocity measurements were taken on 22 irradiated AISI 304 (annealed) miniature tensile-creep specimens. Irradiation temperatures are estimated to fall within 700°F to 1100°F . Neutron fluences are estimated to fall within 1×10^{19} to 1.1×10^{21} nvt > 1 MeV in a fast flux spectrum. Measurements were made using a through-transmission method. The reference specimen was not thermally aged.

Results are shown as a histogram in Figure 15. Changes in velocity ranged from an increase of 0.27% to a decrease of 0.86%. Two other specimens, which were 25% cold worked, showed increases

Estimated Irradiation Temperature: 700⁰F to 1100⁰F

Estimated Neutron Fluence: 1×10^{19} nvt to 1.1×10^{21} nvt
($E > 1$ MeV)

Pre-Irradiated Annealed 1 hr @ 1950⁰F

FIGURE 15
Distribution of Change-in-Longitudinal-Velocity Measurements for Twenty-Two
EBR-II Irradiated 304 Miniature Tensile-Creep Specimens

of 0.31% and 0.73%. The effects of cold-working and high irradiation temperatures undoubtedly have contributed to changes but to what extent remains undetermined.

Measurements on five unirradiated specimens indicate error due to scatter will be at least 0.2%.

Changes in Elastic Properties

Change-in-density data, necessary for approximating the elastic constants changes, are found in Table V. Also tabulated in Table V are specimens' cross-sectional measurements. When compared to measurements taken before irradiation, swelling can be determined — subject to error related to the remote micrometer measurement method. Swelling on specimen 306LT averaged 0.0003 in. on a cross section.

The apparent effect of irradiation on elastic properties has been calculated for two specimens, 306LT and 3H4LT. Elastic properties for irradiated specimens 306LT and 3H4LT are shown in Table VI.

Sample calculations for specimen 306LT follow:

- shear modulus is given by $G = \rho V_S^2$

where ρ = density, and

V_S = shear velocity

$$\Delta G/G = \Delta \rho/\rho + 2 \Delta V_S/V_S$$

$$\Delta G/G \cong -0.0023 + 2(-0.0126) \cong -2.75\%$$

- bulk modulus is given by $K = \rho V_L^2 - 4/3 V_S^2$

where V_L = longitudinal velocity

TABLE V

SWELLING AND CHANGE IN DENSITY,IRRADIATED 304

Specimen Identi- fication	Rounded Average Dimension Pos. 1&4, in. x 10 ⁻³	Rounded Average Dimension Pos. 2&5, in. x 10 ⁻³	Rounded Average Dimension Pos. 3&6, in. x 10 ⁻³	Corrected Average Swelling Pos. 1-6, in. x 10 ⁻³	Change in Density, $\Delta\rho/\rho$, %
3C1LT	394.0	394.1	393.8	0.08	-0.06
3C2LT	393.9	394.2	394.1	0.20	-0.15
3C3LT	394.6	394.9	394.5	0.30	-0.23
3C4LT	394.6	394.5	394.4	0.16	-0.12
3C5LT	394.6	394.7	394.6	0.31	-0.23
3C6LT	394.5	394.6	394.8	0.30	-0.23
3C7LT	394.4	394.4	394.8	0.40	-0.30
3H4LT	393.6	393.9	394.0	0.08	-0.06
3H5LT	394.5	394.2	394.3	0.10	-0.07
3H7LT	394.5	394.0	394.3	0	0
3U0LT (Unirr- adiated)	394.5	394.3	394.4		

TABLE VI
ELASTIC PROPERTIES FOR UNIRRADIATED 304 STEEL,
AND APPARENT CHANGES IN ELASTIC PROPERTIES
FOR TWO IRRADIATED 304 SPECIMENS

Property	Specimen 3C6LT	Specimen 3H4LT
Shear Velocity, V_S , cm/sec $\times 10^5$	3.14	3.14
Longitudinal Velocity, V_L , cm/sec $\times 10^5$	5.74	5.74
Density, ρ , gm/cm ³	7.93	7.93
Shear Modulus, G , dynes/cm ² $\times 10^{11}$	7.81	7.81
Bulk Modulus, K , dynes/cm ² $\times 10^{11}$	15.70	15.70
Poisson's Ratio, σ	0.286	0.286
Young's Modulus, E , dynes/cm ² $\times 10^{11}$	20.10	20.10
ΔV_S , cm/sec $\times 10^5$	-0.040	-0.007
$\Delta V_S/V_S$, %	-1.26	+0.23
ΔV_L , cm/sec $\times 10^5$	-0.030	-0.006
$\Delta V_L/V_L$, %	-0.51	+0.11
$\Delta \rho$, gm/cm ³	-0.018	-0.005
$\Delta \rho/\rho$, %	-0.23	-0.06
ΔG , dynes/cm ² $\times 10^{11}$	-0.215	+0.031
$\Delta G/G$, %	-2.75	+0.40
ΔK , dynes/cm ² $\times 10^{11}$	-0.032	-0.017
$\Delta K/K$, %	-0.26	-0.11
$\Delta \sigma$	+0.0045	+0.0007
$\Delta \sigma/\sigma$, %	+1.58	+0.26
ΔE , dynes/cm ² $\times 10^{11}$	-0.483	-0.094
$\Delta E/E$, %	-2.4	-0.47

$$\frac{\Delta K}{K} = \frac{\Delta \rho}{\rho} + 2 \frac{V_L \Delta V_L}{V_L^2 - 4/3 V_S^2} - \frac{8}{3} \frac{V_S \Delta V_S}{V_L^2 - 4/3 V_S^2}$$

$$\Delta K/K = \Delta \rho/\rho + \Delta V_L (0.578 \times 10^{-5}) - \Delta V_S (0.422 \times 10^{-5})$$

$$\Delta K/K \cong -0.0023 - 0.017 + 0.0167 \cong -0.26\%$$

• Poisson's ratio is given by $\sigma = \frac{V_L^2 - 2V_S^2}{2(V_L^2 - V_S^2)}$

$$\frac{\Delta \sigma}{\sigma} = \frac{2V_S V_L}{(V_L^2 - 2V_S^2)(V_L^2 - V_S^2)} (V_S \Delta V_L - V_L \Delta V_S)$$

$$\Delta \sigma/\sigma = 0.367 \times 10^{-5} \Delta V_L - 0.672 \times 10^{-5} \Delta V_S$$

$$\Delta \sigma/\sigma \cong -0.0108 + 0.0266 \cong + 1.58\%$$

• Young's modulus is given by $E = V_S^2 \frac{3V_L^2 - 4V_S^2}{V_L^2 - V_S^2}$

$$\frac{\Delta E}{E} = \frac{\Delta \rho}{\rho} - \left[\frac{2V_S V_L^2}{(V_L^2 - V_S^2)(3V_L^2 - 4V_S^2)} - \frac{2}{V_S} \right] \Delta V_S + \left[\frac{2V_L V_S^2}{(V_L^2 - V_S^2)(3V_L^2 - 4V_S^2)} \right] \Delta V_L$$

$$\Delta E/E = \Delta \rho/\rho + \Delta V_S (0.488 \times 10^{-5}) + \Delta V_L (0.082 \times 10^{-5})$$

$$\Delta E/E \cong -0.0023 - 0.0193 - 0.0024 \cong -2.4\%$$

Changes in Critical-Angle-Method Signal Amplitude

A tabulation of measurements is shown in Table VII. A final data point for a specimen is the average of several RF amplitude measurements and several integrated-scan measurements. Normalizing

TABLE-VII

CHANGE IN CRITICAL ANGLE SIGNAL AMPLITUDE IN IRRADIATED 304

Specimen Identi- fication	Average of Four RF Meas- urements Sides 1&3, v_1	Average of Four Integra- ted Scans Sides 1,2,3,4, v_2	Average of Two Integra- ted Scans Sides 1&3, v_3	Normal- ized Ave. of Four RF Msmts. ($\times 4$), ΔA_1	Normal- ized Ave. of Four Int. Scans ($\times \frac{71.12}{5.65}$), ΔA_2	Normal- ized Ave. of Two Int. Scans ($\times \frac{71.12}{25.96}$), ΔA_3	Weighted Ave. of $\Delta A_1, \Delta A_2,$ & $\Delta A_3,$ ΔA_{CA}
3C1LT	1.52	0.70	2.89	6.08	8.82	7.94	7.92
3C2LT	3.50	1.24	5.80	14.00	17.65	15.95	16.32
3C3LT	10.60	3.06	13.46	42.40	38.60	37.00	39.15
3C4LT	12.90	4.32	20.86	51.60	54.30	57.26	54.37
3C5LT	16.92	5.62	23.96	67.68	71.00	65.80	68.87
3C6LT	19.02	6.22	28.16	76.08	78.30	77.10	77.45
3C7LT	17.78	5.65	25.96	71.12	71.12	71.12	71.12
3H4LT	- 3.20	- 0.87	- 4.09	-12.80	-11.00	-11.24	-11.51
3H5LT	2.28	0.21	1.06	9.12	2.65	2.91	4.34
3H7LT	- 2.85	- 0.70	- 3.47	-11.40	- 8.32	- 9.52	- 9.64

is performed to make the several measurements compatible with each other. Final data points are plotted in Figure 16.

From the curves in Figure 16, the following observations are noted:

- Neutron irradiation has caused an increase in critical-angle-method signal ΔA_{CA} in the approximate amount of 35 (arbitrary units) per decade in the range of 10^{18} nvt to 2×10^{20} nvt (>1 MeV) for low temperature reactor irradiations ($T \cong 140$ °F to 350 °F). Ignoring anomalies, the relationship may be expressed as follows:

$$\Delta A_{CA} \cong \Delta A_{CA}^i + 35 \log \phi t / 10^{18}$$

where $\Delta A_{CA}^i \cong 5$ (arbitrary units), and

ϕt = neutron fluence >1 MeV.

- Change in critical-angle-method signal amplitude appears to be in the increasing direction for high temperature reactor irradiations ($T \cong 610$ °F to 680 °F). Changes may have been in the opposite direction at lower fluences than used in this study.
- If a sufficient number of measurements can be made so as to reduce scatter error, the radiation damage on low temperature irradiated 304 can be detected at exposures as low as about 2×10^{18} nvt >1 MeV.
- Specimens receiving the highest exposure, for both high and low temperature irradiated groups, have changes less than

FIGURE 16
Change in Ultrasonic Critical-Angle-Method Signal Amplitude for Irradiated 304

would be expected from their curves' trends.

Scatter and Error

Scatter is defined as a possible variation in the measured ultrasonic property due to material property variations existing before irradiation. Measurements were not taken before irradiation. Scatter was determined from measurements taken on unirradiated control specimens. Some control specimens were thermally aged for a three week period: one each at temperatures from 200 °F through 800 °F in 100 °F increments. Velocities and critical-angle-method changes due to thermal aging were judged to be negligible. See Table VIII for a listing of measurements taken on control specimens.

Scatter was exceptionally severe in the 304 specimens, much more severe than in the low alloy steel specimens.⁽³⁾ Scatter was so severe in the 304 specimens as to render single position measurements largely meaningless. Several measurements were necessary per specimen to obtain the curves shown in this report.

Measurements taken on control specimens determined how much correction should be given each measurement as an allowance for the reference specimen, 3UOLT, not being an average representation for all the control specimens. Shear velocity measurements were individually corrected depending on the measurements taken on control specimens which came from donor plate positions adjacent to those of the irradiated specimens. Longitudinal velocity measurements on irradiated specimens were each given the same correction (+0.033%

TABLE-VIII

CHANGES IN SHEAR VELOCITY, LONGITUDINAL VELOCITY, AND
CRITICAL ANGLE SIGNAL AMPLITUDE FOR 304
CONTROL SPECIMENS

Speci- men Identi- fication	Heat Treat- ment for 3-Weeks, °F	Change in Shear Velocity, $\Delta V_S/V_S$ (b,c)			Change in Longitudinal Velocity, $\Delta V_L/V_L$ (b,c)		
		Posi- tion 1-4, %	Posi- tion 2-5, %	Posi- tion 3-6, %	Ave. of Posi- tions 1&4, %	Ave. of Posi- tions 2&5, %	Ave. of Posi- tions 3&6, %
3LT1	As Ma- chined	-.016	+.113	+.118	-.045	+.022	+.029
3NOLT	Stress Relief Only	+.001	+.032	+.197	-.034	+.056	-.002
3X3LT	Stress Relief Only	+.081	+.043	+.158	-.002	+.128	+.080
3X6LT	Stress Relief Only	+.230	+.125	+.148	+.138	+.188	+.102
3LT5	Stress Relief Only	+.057	+.178	+.121	-.027	+.060	-.005
3UOLT ^(a)	200	0	0	0	0	0	0
3LT2	300	-.016	+.061	+.093	-.023	+.015	+.018
3LT3	400	+.060	-.016	+.151	+.001	+.002	+.040
3X1LT	500	-.007	+.059	+.140	-.019	+.047	+.054
3X2LT	600	+.072	+.065	+.082	-.027	+.045	+.024
3AOLT	700	+.057	+.013	+.164	-.038	+.013	+.020
3LT4	800	+.169	+.218	+.208	+.044	+.101	+.097

TABLE VIII (Continued)

Specimen Identi- fication	Critical Angle Signal Amplitude, ΔA_{CA} (b,c,d)		
	Average of Eight RF Measurements, v_1	Average of Four Integ- rated Scans, v_2	Normalized & Weighted Ave. of v_1 & v_2 ($\times \frac{71.12}{5.65}$), ΔA_{CA}
3LT1	+0.285	+0.266	+3.43
3NOLT	+0.110	+0.090	+1.22
3X3LT	-0.130		-1.64
3X6LT	+0.172		+2.16
3LT5	+0.180		+2.26
3UOLT ^(a)	0	0	0
3LT2	-0.365	-0.300	-4.05
3LT3	-0.255	-0.305	-3.63
3X1LT	+0.530	+0.509	+6.50
3X2LT	-0.252	+0.032	-0.79
3AOLT	-0.155	-0.242	-2.69
3LT4	-0.135	-0.709	-6.51

Notes: (a) Specimen 3UOLT used as reference for all measurements.

(b) A + reading designates an increase in velocity or signal amplitude relative to the same measurement at a similar position on 3UOLT.

(c) A - reading designates a decrease in velocity or signal amplitude relative to the same measurement at a similar position on 3UOLT.

(d) v_1, v_2 , = arbitrary units of voltage amplitude.

subtracted from the average of six readings for each specimen).

No corrections were given critical-angle-method measurements.

Measurements on the control specimens also determined scatter limits. Those control specimens which had the greatest deviation in the positive and in the negative directions relative to the reference specimen, 3UOLT, determined the scatter limits.

Error was determined through a series of repetitive measurements, both micrometer measurements of specimen cross-section dimensions and ultrasonic measurements. An additional error contribution was allowed for method, water temperature variations, etc. Errors are: $\Delta V_S/V_S = \pm 0.1\%$, $\Delta V_L/V_L = \pm 0.15\%$, $\Delta A_{CA} = \pm 2.4$ (normalized scale).

DISCUSSION

General

A trend for decreasing ultrasonic propagation velocities has been shown to exist as a function of increasing neutron fluence. The critical-angle-method curves are seen to approximate the shapes of the ΔV curves. Changes in velocity are the probable cause of the changes in critical angle signal although changes in density and attenuation will also contribute.

Changes in velocity relate to changes in elastic properties through the equations of motion in an elastic medium. Irradiated 304 can only approximately be considered as an isotropic, homogeneous, elastic material. Consequently, the results given in the previous section must also be approximate and useful more as an indication

of trends rather than as absolute quantities.

The measured changes are approximately double the changes measured on low alloy steels for identical temperatures and neutron fluences.⁽³⁾ Causes of the larger changes in 304 are undetermined although, in addition to composition and atomic crystalline structure differences, it is noted that 304 had been solution annealed prior to irradiation whereas the low alloy steels had been quenched and tempered.

Shapes of 304 and low alloy steel curves are about the same with some exceptions. The most notable difference is the absence of a reduction in slope in the C4 to C5 specimens region for 304. Another difference appears on the 304 H7 data point for ΔA_{CA} , a data point displaced unexpectedly far in the increased velocity direction. Some of the differences in curve shapes, again, probably relate to the metallurgical differences between the steels.

Cause of the anomalous ΔA_{CA} data point for 3H7LT is undetermined. It need not be surprising that the ΔA_{CA} curves are somewhat different from the ΔV curves, however, since the critical-angle method measures changes in surface properties whereas the ΔV methods measure changes in properties extending through most of the specimen's cross section.

Effects of Irradiation Temperature

A listing of irradiation temperatures is seen in Table II. It is seen that calculated temperatures jumped sharply starting

with the C5 specimen relative to the C4 specimen; and a further increase was seen by the C7 specimen relative to the C6 specimen. Although the increased temperatures are the apparent cause of the changed trend of ΔV measurements for the low alloy steel C5 specimens,⁽³⁾ the ΔV measurements for 304 remained on a smooth trend curve. Irradiation damage in 304 steel is apparently not quite as sensitive to irradiation temperature as it is in A302-B or A542-B — at least up to the C7 specimen's temperatures, about 285 °F to 376 °F.

The C7 specimen was affected for 304 in a manner similar to that of the low alloy steels. A significantly higher irradiation temperature for the C7 specimens relative to the C6 specimens is the probable cause of the change in trend from C6 to C7.

The three specimens irradiated at high temperatures are much less affected than are the seven specimens irradiated at low temperatures. Apparently, damage recovery is accelerated at some temperature lying between about 300 °F and 680 °F. Whether recovery occurs somewhat linearly as a function of temperature or in jumps has not been determined during this study.

Measurements on thermally aged control specimens (see Table VIII) showed little change in velocities as a function of temperature only. The combined effects of temperature and irradiation, however, are not too well defined. The increased velocities in some high temperature irradiated specimens could be primarily as a result of

heating rather than irradiation. On the other hand, the increased velocities could also be as a result of thermally activated irradiation damage recovery mechanisms affecting one contributor to elastic properties changes but not affecting another opposing contributor.

Effects of Variation in Neutron Energy Spectrum

As seen in Table I, the C5, C6, C7, H4, H5, and H7 specimens all were irradiated in a "harder" neutron energy spectrum than were the C1, C2, C3, and C4 specimens. A slight reduction in slope of the low temperature ΔV & ΔA_{CA} curves could feasibly be partly as a result of the low thermal/fast neutrons ratio for the C5, C6, and C7 specimens. In other words, there may be a noticeable contribution to damage, as measured by ultrasonic ΔV methods, from lower energy (less than 1 MeV) neutrons bombardment. Much of the mechanical damage to austenitic steels is thought to result from low energy neutron bombardment; capture of thermal neutrons by some elements results in gas generation and accumulation which tends to be more severe at grain boundaries.^(6,7) It appears more likely, however, for the work described herein, that the detected damage is as a consequence of atomic displacements rather than transmutations as the gas concentration is quite low and not apt to significantly affect elastic properties.

Causes of Changes in Velocities

Several mechanisms are thought to contribute to elastic properties changes. One of them, the dislocations density and

and damping mechanism first described at megahertz frequencies by Granato and Lücke,^(8,9) stands apart from the others. Under this mechanism, velocities might be expected to first increase due to radiation defects interactions with dislocations, then decrease as dislocation density increases at higher fluences due to collapse of clusters of defects, and then once again to increase as dislocations become so dense as to interact with each other.

Other mechanisms seem to be related to each other. Included are effects on elastic constants due to:

- interstitials and vacancies, calculated by Dienes using an interatomic potential energy approach;⁽¹⁰⁾
- spherical holes, one treatment of which has been done by MacKenzie using classical elasticity theory;⁽¹¹⁾ and
- multiple scatterers, as described by Truett and colleagues.^(12,13)

Although some conflicting effects might be predicted, in general one would expect reduced velocities if reduced-atomic-density zones predominated over increased-atomic-density zones. Reduced-atomic-density zones might predominate at the temperatures used in this study where interstitials are more mobile than their vacancy counterparts⁽¹⁴⁾ and hence more likely to anneal-out to some location where their presence would be relatively unfelt such as at grain boundaries, surfaces, and other sinks.

Relationship of Changes in Ultrasonic Velocities to Change in Ductility

Ultrasonic methods appear to be feasible for detection of

radiation damage at lower irradiation temperatures. As shown in Figure 17, changes in ΔV_S (or ΔV_L or ΔA_{CA}) appears indirectly correlative to degraded ductility as measured by total elongation. At higher irradiation temperatures, however, ultrasonic changes become less, but degraded ductility becomes greater. Other measurements, as shown in Figure 15, indicated there may be significant velocity changes at irradiation temperatures higher than used in this study. Higher accuracy measurements, such as would result if pre-irradiation measurements are taken, will be necessary to determine effectiveness of ultrasonic measurements of high temperature irradiation damage. In addition, the effects of high temperature thermal aging on ultrasonic velocities should be determined for higher temperatures and longer periods than those shown in Table VIII.

Suggested Applications

Ultrasonic methods appear feasible for assessment of atomic displacements related damage in low temperature irradiated austenitic steels. If irradiated at higher temperatures or if annealing takes place after irradiation, accurate damage assessment may be possible only when pre-irradiation measurements are taken such as might be done on surveillance specimens.

Due to a high sensitivity to temperature as an irradiation parameter, ultrasonic methods appear to be useful for laboratory studies on radiation damage and recovery.

FIGURE 17
Effects of Neutron Irradiation on Ultrasonic Shear Velocity
and on Room Temperature Ductility for Annealed 304 Steel

ACKNOWLEDGEMENTS

The work described in this report was done under the sponsorship of Fuels and Materials Branch of the USAEC Division of Reactor Development and Technology.

Numerous helpful comments by several Pacific Northwest Laboratory staff members are gratefully acknowledged. Also acknowledged are the services of C. E. Christensen, PNL, NDT Methods Development, who did most of the specimens handling and measurements, and developed part of the electronic instrumentation used during the study.

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