Advanced Technology Laboratories
A Division of American-Standard

INVESTIGATION OF
THERMAL-STRESS-FATIGUE BEHAVIOR
OF STAINLESS STEELS

Prepared under
AEC Contract AT(04-3)-250
Project Agreement No. 11
for the
Joint U.S.-Euratom Research and Development Board

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INVESTIGATION OF
THERMAL-STRESS-FATIGUE BEHAVIOR
OF STAINLESS STEELS
Quarterly Progress Report No. 8
ATL Job 121200
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INTRODUCTION

The United States and the European Atomic Energy Community (Euratom), on May 29, and June 18, 1958, signed an agreement which provides a basis for cooperation in programs for the advancement of the peaceful applications of atomic energy. This agreement, in part, provides for the establishment of the Joint U.S.-Euratom research and development program which is aimed at reactors to be constructed in Europe under the Joint Program.

The work described in this report represents the Joint U.S.-Euratom effort which is in keeping with the spirit of cooperation in contributing to the common good by the sharing of scientific and technical information and minimizing the duplication of effort by the limited pool of technical talent available in western Europe and the United States.
SUMMARY

Phase I of the program on thermal-stress-fatigue (TSF) testing of 304 and 304-L austenitic stainless steels has been completed. The topical report covering this phase has nearly been completed.

The testing of ferritic steel (ASTM Type A302, Grade B) and of martensitic stainless steel (Type 403) has been approximately 50% completed, with full completion on A302B contemplated for the next quarter.

During this quarter, TSF and conventional strain-cycling fatigue (SCF) were performed on A302B and 403 steels. A new graphical technique was developed which simplifies the work required to obtain plastic strain range, the variable found to be entirely independent in TSF. The new method also allows greater accuracy in obtaining this parameter.
PRINCIPAL INVESTIGATORS

Investigators on the project include K. E. Horton, Project Leader, and R. S. Stewart, Section Supervisor.

STATEMENT OF PROBLEM

The thermal-stress-fatigue behavior of reactor materials, in particular stainless steels, cannot be predicted by extrapolation of known data such as conventional fatigue data. Because thermal fatigue may be a major consideration in reactor core and vessel design, suitable engineering data are needed. The objective of this program is to obtain thermal-stress-fatigue data on various types of stainless steels for the conditions that may be encountered in reactor operation and if possible to separate the effects of the various environmental and metallurgical variables. The isolation of variables should allow a theoretical treatment of data that would eventually lead to the formalization of generalized equations for predicting thermal-stress-fatigue life for any reactor conditions.

PROGRESS OCTOBER-DECEMBER 1963

A. Type A302B Ferritic Steel

The testing of this steel was continued during the quarter. The previous difficulty of oxidation during tests performed in argon and vacuum were overcome by the rebuilding of one TSF tester into a high-vacuum setup. The rebuilt chamber is shown in Figure 1. Although this chamber easily evacuates to $1 \times 10^{-6}$ mm Hg, a zirconium gettering filament has also been incorporated to preferentially capture any oxygen that may still be present in the chamber.

The very low strength of A302B at elevated temperatures has dictated a maximum temperature of 500°C as suitable for TSF testing. As a result, a significant portion of the tests are of the high-cycles-to-failure, low-plastic-strain-range ($\Delta \epsilon_p$) type.

In the range of $\Delta \epsilon_p$'s below $1 \times 10^{-3}$, an appreciable error is graphically introduced in calculating $\Delta \epsilon_p$ by the method formerly used. That is, when the constrained measurement gage length ($l_o'$) movement (recorded versus time) is graphically subtracted from the unconstrained $l_o'$ movement (recorded versus time), a slight


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The misplacement of the time axis before subtraction can lead to dismaying errors in the calculated value of $\Delta \varepsilon_p$.

This method is, of course, theoretically correct for determining $\Delta \varepsilon_p$ within $l_0'$, even if plastic strain also occurs outside of $l_0'$. The method assumes that $\Delta \varepsilon_p$ is constant within $l_0'$, and for a $\frac{1}{2}$-inch $l_0'$ this assumption is nearly correct. It perhaps should be mentioned that $\Delta \varepsilon_p$ was obtained by plotting the remainder of the above subtraction process versus load (both are known as a function of time), and since the width of the resulting hysteresis loop is directly proportional to plastic movement, $\delta_p$, it was necessary only to divide $\delta_p$ by $l_0'$ to obtain $\Delta \varepsilon_p$.

An alternative way to obtain $\Delta \varepsilon_p$ is to record the following measurements on an X-Y recorder: 1) temperature versus unconstrained movement of $l_0'$, 2) temperature versus load, and 3) temperature versus constrained movement of $l_0'$. Such recordings are illustrated in Figure 2. This method differs from that proposed by others in that the additional step 3 is included which, since it allows for plastic strain outside the measurement gage length, allows the practically valid assumption of $\Delta \varepsilon_p$ being constant within $l_0'$. The advantage of having $\Delta \varepsilon_p$ constant within $l_0'$ is clearly seen in equation 3, which is here derived.

$$\delta_p = \delta_1 + \delta_2 = l_1 \Delta \varepsilon_p^1 + l_2 \Delta \varepsilon_p^2 + l_0' \Delta \varepsilon_p^3 + l_{o'} \Delta \varepsilon_p^4$$

$$\begin{align*}
\delta_p &= l_1 \Delta \varepsilon_p^1 + l_2 \Delta \varepsilon_p^2 \\
&= l_0' (\Delta \varepsilon_p^1 + \Delta \varepsilon_p^2) \\
&= \sum_3^n \Delta \varepsilon_p^3 = \frac{l_0' (\Delta \varepsilon_p^1 + \Delta \varepsilon_p^2)}{l_0'}
\end{align*}$$

That is, $\delta_p$ is divided by a constant length, $l_0'$, which is easily measurable.

An even more direct graphical method than that shown in Figure 2 is available to obtain $\Delta \varepsilon_p$. With reference to Figure 3, it is seen that $\delta_1$ would be the value of $\delta_p$ if we neglect

* Prof. A. Carden, University of Alabama, private communication, 1963.
** See list of symbols, page
the fact that this $\delta$ is obtained by measuring the instantaneous length of $l_0'$ at two different temperatures (where $P = 0$). The addition of $\delta_2$ to $\delta_1$ corrects for the difference in temperature at zero loads. The value of $\Delta \epsilon_p$ is found by algebraically adding $\delta_1$ and $\delta_2$ and dividing the sum by $l_0'$.

It may be argued that if $\Delta \epsilon_p$ is not constant within $l_0'$ (that is, if temperature is not uniform over $l_0'$), the two graphical methods just discussed will be in error proportional to the variation in temperature gradient along $l_0'$. With the experimental equipment and specimen design used by ATL, the maximum variation of $T$ along $l_0'$ is approximately $20^\circ$C, a value sufficiently small to disallow a significant strength (or ductility) variation along $l_0'$. On the other hand, the variation in $T$ along the total geometric gage length can be as great as $400^\circ$C. Clearly, this magnitude will greatly vary strength along the length; it thus becomes necessary to correct for this variation by dividing $\delta_p$ measured over the geometric length by a completely fictitious length to get $\Delta \epsilon_p$ maximum. The divisor is fictitious in the sense that total plastic movement of the specimen was measured and this value must be divided by a length to give $\Delta \epsilon_p$ such that if the uniform plastic movement have been measured over this length and divided by this length the same value of $\Delta \epsilon_p$ would result.

The graphical technique illustrated in Figure 3 is very rapid and has been adopted for use on this program in the future. With this method it is easy to ascertain values of $\Delta \epsilon_p$ as small as $5 \times 10^{-5}$, whereas the older method of calculating $\Delta \epsilon_p$ was limited to values of $\Delta \epsilon_p$ in the $5 \times 10^{-4}$ range and above. The difficulty of calculating small values of $\Delta \epsilon_p$ by the old method, and the oxidation problem noted earlier, were responsible for the data scatter mentioned in the previous quarterly progress report.† The new‡‡ graph of $\Delta \epsilon_p$ versus $N$ is shown in Figure 4. There are two noteworthy manifestations on this graph: 1) the agreement between TSF failure and conventional fatigue (strain-cycling) failure data, and 2) the graphical inference of only one line of one slope for describing both stable and unstable (buckling) failure. This observation suggests

* Length of minimum cross-sectional area.
** The direct readout of movement is possible to $2 \times 10^{-5} \pm 1 \times 10^{-5}$.
‡‡ To replace Figure 9 of Quarterly Progress Report No. 7, ATL-A-139.

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that instability failure is a metallurgical phenomenon and not a phenomenon dependent on specimen geometry. Whether or not there is any difference in the curves for unstable and stable failures is not certain; however, there is a substantial difference between these two failure curves in 304-L stainless steel. *

In the test program just completed on 304-L stainless, an unsuccessful attempt was made to distinguish between stable and unstable fatigue life at a given level of plastic strain range. Since this attempt was made by varying the specimen wall thickness, it was assumed that the thinner walled specimens were geometrically more unstable. Actually, the test conditions may have been such that the specimens were metallurgically unstable in that a net plastic strain occurred in one direction (during compression at elevated temperatures). The longitudinal temperature gradient present would transform a cylindrical shape into a barrel shape, which would certainly be conducive to unstable failure as illustrated in Figure 5. Whatever the cause for instability failure, it is uncertain if it is experimentally possible to cause TSF failure in 304-L of any specimen design in the low cycle-to-failure range and not get an instability failure. The same uncertainty exists for A302B ferritic steel. **

The structures of the specimens after testing are shown in Figures 7 through 11; Figure 6 is included to allow comparison with the untested structure. In these photomicrographs, it is seen that the low-cycles-to-failure structure (Figure 7) is different from both the as-reversed and the high-cycles-to-failure structures. The structure shown in Figure 7 was obtained by TSF testing between 800 and 200°C. This same structure can also be obtained by water quenching A302B from 927°C, as shown in Figure 12. This structure is probably upper bainite, although it is very soft due to the low carbon content of A302B.


** To resolve this uncertainty, it is planned to reverse the stress-temperature relationship. That is, specimens will be in tension at the high temperature and in compression at the low temperature. This end will be accomplished with slight modification of existing strain-cycling equipment.
Of interest is the completely different structure obtained by TSF cycling a specimen identical to that shown in Figure 7 but without a hold time at temperature extremes. This new structure, shown in Figure 13, is probably due to insufficient time for austenitizing; hence little if any bainite is formed.

The significantly different structures present in TSF-tested A302B do not alter the position of the failure curve shown in Figure 4. While this may appear remarkable, it should be remembered that all the structures exhibited by A302B are merely different arrangements of ferrite and cementite.

The oxidation that took place during the air tests and vacuum tests conducted before the rebuilding of the vacuum test chamber prohibits determination of the fracture path. However, the oxide formation in a fatigue crack does serve a useful purpose in that it allows a rough estimate of the fracture rate. To illustrate, Figures 14 and 15 show a uniform gradation in oxide thickness in the fatigue crack, which suggests a uniform rate of crack propagation. Furthermore, comparison of the maximum thickness of the fracture-path oxide with the thickness of the surface oxide indicates that fracture did not start until about two-thirds of fatigue life was reached.

B. Type 403 Martensitic Stainless Steel

The failure curve for the data obtained to date on this steel is plotted in Figure 16. When this graph is compared to that in Figure 4, it is seen that Type 403 steel is more fatigue resistant than A302B ferritic steel. Both graphs have similar slopes, however. The structures of 403 steel in various conditions are illustrated in Figures 17, 18, 19, and 20, for comparison with TSF-tested structures. It can be seen that the quenching portion of the heat treatment produces a martensitic structure, which, of course, is tempered by reheating to \( \sim 650°C \). The structures after fatigue testing are shown in Figures 21 through 24. Figure 21 shows that complete tempering has occurred by cycling from \( 700°C \) with 1-minute hold at this temperature. Even without the 1-minute hold at \( 700°C \) during TSF cycling from \( 700°C \), tempering is comparable to a 24-hour temper at \( 645°C \), as seen by comparing Figures 22 and 20. TSF cycling or strain cycling at lower temperatures does not change the tempered martensitic structure originally present except perhaps for a slight further tempering, as shown by comparing Figures 23 and 24 with Figures 19 and 20. The type of fracture paths is difficult to ascertain due to the fine-grain structures present.
Since many tests remain to be performed on 403 steel, discussion of its TSF characteristics is premature. However, there is one noteworthy early conclusion: TSF and SCF failures are indistinguishable on the plot of $\Delta \varepsilon_p$ versus $N$. On the other hand, there is an appreciable difference between TSF and SCF data points on a plot of stress versus $N$, as shown in Figure 25. This state of affairs suggests that $\Delta \varepsilon_p$ is the independent test variable.

PLANS FOR FUTURE WORK

Testing of A302B ferritic pressure-vessel steel will be completed during the next quarter. Prior to completion of these tests, a few preliminary tests will be performed on Croloy 2$\frac{1}{4}$ steel specimens to determine if the specimen design and fabrication result in satisfactory performance. The Croloy specimens are a composite, welded structure in which failure could occur at the weld regions of unsatisfactory specimens. These early tests on Croloy 2$\frac{1}{4}$ will insure full-scale testing of this material after completion of A302B testing.

A few TSF tests will be run in conjunction with ultrasonic testing for fatigue cracks. These tests will permit determination of rate of fatigue-crack growth and time for crack initiation.

CONCLUSIONS

1) A significant improvement has been made in the technique for obtaining $\Delta \varepsilon_p$. The new method is particularly useful in the high-cycles-to-failure tests, permitting accurate measurement of plastic strains as small as 50 microinches per inch.

2) There is only one fatigue-failure curve on graphs of $\Delta \varepsilon_p$ versus $N$ for both pure fatigue and instability failures of A302B ferritic pressure-vessel steel and 403 martensitic stainless steel. This is in direct contrast to the results on 304-L stainless steel and suggests that instability failure is a metallurgical phenomenon that is not geometrically dependent on the specimen design.

3) Failures by SCF and TSF are indistinguishable on graphs of $\Delta \varepsilon_p$ versus $N$, indicating that $\Delta \varepsilon_p$ is the independent variable.
LIST OF SYMBOLS

\( f \) = effective gage length = \( \alpha \Delta T/\delta' \) (inches)
\( f'_{o} \) = measurement gage length (inches)
\( N \) = cycles to failure
\( P \) = load
\( T \) = temperature (°C)
\( \alpha \) = coefficient of thermal expansion (in./in.-°C)
\( \delta \) = movement of \( f'_{o} \) (inches)
\( \delta' \) = movement of \( f \) (inches)
\( \delta_p \) = plastic movement
\( \Delta \varepsilon_p \) = plastic strain range per cycle (inches)
\( \Delta \sigma \) = stress change per cycle (tension plus compression) (psi)
REPORTS ISSUED SINCE INCEPTION OF PROGRAM

Quarterly Technical Progress Report, April-June 1962, ATL-A-131, EURAEC 335
Quarterly Technical Progress Report, July-September 1963, ATL-A-139, EURAEC 841

* Report should have been dated 1963.
TABLE I
PROPOSED TESTING PROGRAM

I.  AUSTENITIC STAINLESS STEEL
   A.  TYPE 304-L
   B.  TYPE 304
   Same as "I"
II. FITRITIC STAINLESS STEEL
    A.  TYPE 403
    Same as "I"
III. MARTENSITIC STAINLESS STEEL
     A.  TYPE 403
     Same as "I"
     A.  TYPE 403
    A.  A302B
    Same as "I"
    Same as "I"
    B.  CROLOY 2 1/2
    C.  INCOLOY
    D.  A302B
    Same as "I"
    Same as "I"
    Same as "I"
IV. OTHER FERROUS ALLOYS
    A.  TYPE 403
    Same as "I"
    Same as "I"
    Same as "I"
    Same as "I"
    Same as "I"
V.  NON-FERROUS ALLOYS
    A.  SAME AS "I"
    Same as "I"

1.  Annealed (Small Grain)
2.  Annealed (Large Grain)
3.  Cold Worked (As Received)
4.  Heat Treated (Tempered Martensite)
   Same as "I"
   Same as "I"
   Same as "I"

a.  Air Tests
b.  Argon Tests
c.  Vacuum Tests
   Same as "a"
   Same as "a"

1.  20-MIL WALL
2.  30-MIL WALL
3.  40-MIL WALL
   Same as "I"
   Same as "I"

(a-d) TSF

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<th>Temperature of Tests</th>
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<td>a</td>
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<td>b</td>
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<td>c</td>
<td>600-200</td>
</tr>
<tr>
<td>d</td>
<td>700-100</td>
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See right for delineation of a) through f) above.

NOTE: All temperatures in °C.
HIGH-VACUUM TSF SETUP

FIGURE 1
NOTE: See List of Symbols, page 9, for definitions of terms.

**STEP 1** - \( T \) vs \( \delta \) unconstrained measured over \( l_0' \)

\[ T_1 \quad (\text{unconstrained}) \rightarrow \]

**STEP 2** - \( P \) vs \( T \), specimen constrained

\[ T_4 > T_3 \]

\[ T_4 - T_3 \approx \Delta \varepsilon_{p_1} + \Delta \varepsilon_{p_2} + \Delta \varepsilon_{p_3} = \delta_1. \]

**STEP 3** - \( P \) vs \( \delta \) constrained measured over \( l_0' \)

\[ \delta \] (constrained) \rightarrow

**STEP 4**

\[ \Delta \varepsilon_{p_3} (\text{in} \ l_0') = \frac{\delta_1 + \delta_2}{l_0}. \]

*GRAPHICAL METHOD OF OBTAINING \( \Delta \varepsilon_p \) MAXIMUM*

**FIGURE 2**
NOTE: See List of Symbols, page 9, for definitions of terms.

\[ \Delta \epsilon_p = \frac{\epsilon_p}{\epsilon_0} \]

**a.** \( \epsilon_0 \) under more than 100% constraint

**b.** \( \epsilon_0 \) under less than 100% constraint

**GRAPHICAL METHOD OF OBTAINING** \( \Delta \epsilon_p \)**

**CURVES RECORDED DIRECTLY ON X-Y RECORDER**

**FIGURE 3**
NOTES:
TESTS IN AIR AND IN VACUUM.
DATA FROM SHORT MEASUREMENT GAGE LENGTH.
REFERENCED FIGURES ARE THOSE IN THIS REPORT.

PLASTIC STRAIN RANGE VERSUS CYCLES TO FAILURE
FOR A302B FERRITIC PRESSURE-VESSLE STEEL

FIGURE 4
FAILED TSF SPECIMEN
ILLUSTRATING INSTABILITY-TYPE FAILURE

FIGURE 5
AS-RECEIVED (ANNEALED) ASTM TYPE A302B FERRITIC PRESSURE-VESSSEL STEEL
Etched 15 sec in 5% Nital. Hardness: Vickers 26; R_H 19.

FIGURE 6

TSF-TESTED A302B FERRITIC PRESSURE-VESSSEL STEEL
Specimen 251. Test IV. A.1. a, 2. c)–2c

FIGURE 7

* See Table I for explanation of test conditions.
TSF-TESTED A302B FERRITIC PRESSURE-VESSEL STEEL
Specimen 253. Test IV. A.1. a. 2. a)-2b
Etched 80 sec in 1% Nital.

FIGURE 8

SCF-TESTED A302B FERRITIC PRESSURE-VESSEL STEEL
Specimen 287. Test IV. A.1. a. 1. e)-1b
Etched 15 sec in 5% Nital.

FIGURE 9
SCF-TESTED A302B FERRITIC PRESSURE-VESSEL STEEL
Specimen 292. Test IV, A.1, a.1, e)-xa (Test temp. = 400°C)
Etched 10 sec in 5% Nital.

FIGURE 10

TSF-TESTED A302B FERRITIC PRESSURE-VESSEL STEEL
Specimen 275. Test IV, A.1, a.2, c)-4c
Etched 5 sec in 5% Nital.

FIGURE 11
STRUCTURE OF A302B FERRITIC STEEL
WATER QUENCHED FROM 927°C (1700°F)
Etched 7 sec in 5% Nital. Hardness: Vickers 35.6

FIGURE 12

TSF-TESTED A302B FERRITIC PRESSURE-VESSLE STEEL
Specimen 261. Test IV. A.1. a. 2. a)–2c
Etched 7 sec in 5% Nital.

FIGURE 13
TSF-TESTED A302B FERRITIC PRESSURE-VEssel STEEL SHOWING OXIDE FORMATION IN FATIGUE CRACK
Specimen 268. Test IV. A.1. a. 3, c) - 1c
Etched 7 sec in 5% Nital.

FIGURE 14
Fatigue-Crack Oxide

Fracture Path

Surface Oxide

1000x ← Stress → Neg. #5093, 5094

TSF-TESTED A302B FERRITIC PRESSURE-VESSLE STEEL SHOWING OXIDE FORMATION IN FATIGUE CRACK
Specimen 260. Test IV.A.1.a.2.c)-1c
Etched 7 sec in 5% Nital.

FIGURE 15
Data for short measurement gauge length for 433 martensitic stainless steel tested in tempered condition.

Plastic strain range versus cycles to failure.

Legend:
- Instability failure
- Scrap
- Test

Note:
- Referenced figures are those in this report.
Electrolytic Etch: 10% Oxalic Acid.
Hardness: $R_B$ 96-98

Etched 7 seconds in Marbles Reagent

AS-RECEIVED (ANNEALED) TYPE 403 STAINLESS STEEL

FIGURE 17
TYPE 403 STAINLESS STEEL QUENCHED IN HELIUM
FROM 1000°C (1832°F)
Etched in 10-HCl, 3 HNO₃, 100 CH₃OH. Hardness: Rₚ 42-44.

FIGURE 18
100 x Oblique Lighting Neg. #4961

TYPE 403 STAINLESS STEEL QUENCHED IN HELIUM
AND TEMPERED 2 HOURS IN VACUUM AT 649°C (1200°F)
Etched in 10-HCl, 3-HNO₃, 100-CH₃OH. Hardness: Rₖ 20-23

FIGURE 19

100 x Oblique Lighting Neg. #4993

TYPE 403 STAINLESS STEEL, SAME HEAT TREATMENT AS
IN FIGURE 19, PLUS 22 ADDITIONAL HOURS AT 649°C
Same etch as in Figure 19. Hardness: Rₖ 87-92

FIGURE 20
TSF-TESTED 403 STAINLESS STEEL
Specimen 353. Test III. A. 4. a. 1. c)-1d
Etched 5 sec in Marbles Reagent. 1-min hold time at $T_{\text{max}} = 700^\circ\text{C}$

FIGURE 21

TSF-TESTED 403 STAINLESS STEEL
Specimen 365. Test III. A. 4. a. 1. a)-2b
Etched 5 sec on Marbles Reagent. Zero hold time at $T_{\text{max}} = 700^\circ\text{C}$

FIGURE 22
TSF-TESTED 403 STAINLESS STEEL
Specimen 356. Test III. A. 4. a. 1. c) - 1c (Tmax = 600°C)
Etched 10 sec in Marbles Reagent.

FIGURE 23

SCF-TESTED 403 STAINLESS STEEL
Specimen 354. Test III. A. 4. a. 1. e) - 1e (Test temp. = 400°C)
Etched 5 sec in Marbles Reagent.

FIGURE 24
STRESS CHANGE (TENSION PLUS COMPRESSION) PER CYCLE VERSUS CYCLES TO FAILURE FOR 403 STAINLESS STEEL

FIGURE 25
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