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Advanced Technology Laboratories
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MASTER

INVESTIGATION OF
THERMAL-STRESS-FATIGUE BEHAVIOR
OF STAINLESS STEELS

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Special Distribution

INVESTIGATION OF
THERMAL-STRESS-FATIGUE BEHAVIOR
OF STAINLESS STEELS

Quarterly Progress Report No. 7

ATL Job 121200

July-September 1963

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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

The final report on Phase I of the program (austenitic stainless steels) has been started; it is estimated that this topical report will be issued during the next quarter.

During this quarter, testing of Type 304-L austenitic stainless steel was completed. Thermal-stress-fatigue testing was started on ferritic pressure-vessel steel (ASTM type A302, Grade B), and work on this alloy is approximately half completed. Test specimens of Type 403 martensitic stainless steel were machined and the heat treating schedule worked out in anticipation of testing this steel during the next quarter. The machining of test specimens of Croloy 2-1/4 steel was also started.

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 JULY-SEPTEMBER 1963

The tests performed during this quarter are listed in Table I. The primary effort was placed on investigating A302B ferritic pressure-vessel steel, with secondary emphasis on Types 304-L and 403 stainless steels. All work on 304-L austenitic stainless steel is complete, and a topical report covering the TSF behavior of 304 and 304-L is being prepared for publication during the next quarter.

Some welding research has been performed on Croloy 2-1/4 in preparation for the fabrication of test specimens from pipe stock.

A. 304 and 304-L Stainless Steel Test Data

The two types of failure obtained in the TSF and SCF* experiments on 304 and 304-L stainless steel give two distinct failure equations, i.e., one equation for pure fatigue failure and another equation for failure by plastic-strain instability. The data in Figure 1 indicate that these two equations are of the form

$$N^k \Delta \epsilon_p = C^{**} \quad (1)$$

* SCF - strain cycling fatigue, i.e., conventional fatigue.

** See list of symbols at end of report.

and adequately predict failure. It was mentioned in the last quarterly report¹ that plastic instability of tubular columns would probably be dependent on the ratio of wall thickness to mean radius, t/R . However, Figure 1 does not substantiate this expectancy, probably because of the gradual buildup of plastic distortion, which eventually leads to specimen instability. The sequence of photographs in Figure 2 illustrates this gradual buildup of distortion. The dependence of the instability-failure equation on t/R would no doubt be very evident in unidirectional test results.

In fatigue programs^{2,3} there is a tendency to generalize test results and plot tensile test data with fatigue data (plotting tensile failure at one-fourth cycle). When this is done the implication of time independence is obvious. Tensile tests, such as those on stainless steel, that exhibit necking-down should be classified as instability failures and therefore should not agree with conventional fatigue data, let alone TSF data. Instead it is more likely that tensile-test and instability-fatigue data would agree. To test this hypothesis, tensile test data and TSF test data are plotted in Figure 3; the tensile-test strain rate was the same as the TSF strain rate. It is seen that tensile-test failures agree with neither pure-fatigue nor instability failures. This same conclusion can also be drawn about Incoloy, as also shown in Figure 3.

B. Fabrication of Croloy 2-1/4 Specimen

To obtain Croloy 2-1/4 material for TSF testing, it was necessary to purchase seamless pipe of 0.150-inch wall thickness. This pipe is reamed and turned to 0.090-inch wall thickness and is then welded to end bosses of 1018 carbon steel. The assembly before welding is illustrated in Figure 4. The heliarc welding will limit the length of the heat-affected zone such that it will not extend into the gage length.

C. Preparation of 403 Martensitic-Stainless-Steel Specimen

The rather close tolerance required on TSF test specimens could possibly be destroyed by quenching and tempering a finished machined specimen. However, experimental heat treatments on test specimens have shown that essentially no distortion occurs. The structures and hardnesses of 403 steel are shown in

1. See list of references, page 8.

Figures 5 and 6. It is seen from these photomicrographs that long-time tempering treatments have little effect (compare Figures 6a and 6b); hence tempering (e. g., at T') per se will not significantly change the structure of quenched and tempered (at T') 403 stainless steel. Since tempering at 650°C (1200°F) returns the hardness of this steel essentially to that of its annealed condition, it may be desirable to temper at a lower temperature and of course limit the TSF test temperatures to this temperature.

D. A302B Steel Test Program

The ferritic pressure-vessel steel, ASTM Type A302, Grade B, was obtained as plate stock from Hanford and was forged to 1 $\frac{1}{4}$ -inch round bar. The microstructure of annealed bar is shown in Figure 7. The bar, and therefore the TSF test specimens, had a rather heavy inclusion content, as seen in Figure 8. These inclusion stringers run parallel to the specimen axis and have not been found to influence the growth of fatigue cracks to any significant extent.

The results of successful tests on A302B are plotted in Figures 9 and 10. Contrary to the two definite failure lines evident in Figures 1 and 3, only one failure line (for each measurement gage length) is obvious in Figures 9 and 10. Particularly noticeable in Figure 9 is somewhat "gross" data scatter, although the scatterband is less than one order of magnitude. The primary cause for the data scatter is specimen oxidation. The rapid growth of oxide during TSF testing is illustrated in Figures 11 and 12, and it is quite evident that 600°C is too high a temperature for continuous use of A302B in air. When the specimen is run in inert atmosphere, essentially no oxidation occurs, as shown in Figure 13.

The unusual appearance of an "etching gradient" in Figure 13a (see also Figure 11b) is difficult to explain. At first glance, the cause of this gradient appears to be a difference in grain size; however, there is no immediate obvious reason why there should be a grain-size gradient. One plausible explanation for the relatively large grain at the surface of this specimen is secondary recrystallization due to the loss of carbon from the surface (and therefore the elimination of a two-phase structure). The carbon is lost when the ferrite and pearlite are austenitized during TSF cycling. Secondary recrystallization would not occur in the interior of the specimen because of the two-phase structure present. There is appreciably more pearlite in the

interior of the specimen than at the surface, as illustrated in Figure 12b. This explanation also accounts for the slightly lower microhardness of the surface as compared with the interior (ref. Figure 14).

Another observation made on the 800°C maximum test (Figure 13) should be pointed out. A close look at the upper part of Figure 13b reveals an indication of grain elongation. Figure 15 shows this elongation in a more striking way. Normally this elongation would be considered to result from plastic flow during TSF testing; however, the total plastic flow ($N\Delta\epsilon_p$) represented in Figure 15 is equal to 0.388 in./in. as compared to 3.65 in./in. for the equiaxed structure shown in Figure 12. Therefore, the high maximum temperature experienced by the specimens shown in Figures 15 and 16 results in gross specimen distortion (buckling, plastic instability) and therefore grain elongation, even though relatively little total plastic flow has occurred. Conversely, the specimen in Figure 12 did not show a plastic instability failure and therefore no grain elongation. The preliminary conclusion drawn from the foregoing discussion is that plastic flow results in a permanent change in grain shape only if the specimen also exhibits a permanent change in shape.

The difference in hardness between the specimens showing grain elongation and those that did not is probably due to retained austenite. That is, the specimens that were austenitized (partially) during TSF cycling, as were the specimens shown in Figures 13 and 16, retained some of this austenite at room temperature, with a resulting increase in hardness. The specimens that did not austenitize, and incidentally did not manifest grain elongation, were composed of a softer matrix of mainly ferrite plus a little pearlite.

Since many tests remain to be performed on A302B, it is premature to draw further conclusions.

PLANS FOR FUTURE WORK

Testing will continue on A302B ferritic steel with completion expected in three to four months. Simultaneously with the A302B program, testing will be performed on 403 martensitic stainless steel. It is also planned to start fabrication of TSF test specimens of Croloy 2-1/4 such that testing will be able to start in three to four months.

CONCLUSIONS

1) Only one TSF failure equation is evident for A302B, even though there are obviously two failure modes -- pure fatigue (no specimen distortion) and plastic instability (gross specimen deformation).

2) TSF testing of A302B above $\sim 750^{\circ}\text{C}$ allows the formation of retained austenite and thus an increase in hardness. Strain hardening did not cause this hardness increase, since specimens undergoing large total plastic strains did not harden while specimens undergoing a lesser total plastic strain did show a hardness increase (due to retained austenite).

3) The TSF failures of A302B have all been ductile insofar as significant total plastic strain has occurred. Plastic instability results in premature fatigue failure that outwardly appears very ductile. In reality, the specimen failing by instability has undergone relatively little plastic strain compared to what it is capable of undergoing in high-cycle fatigue testing.

4) Changing the wall thickness from 0.020 inch to 0.040 inch did not affect the TSF resistance of 304-L stainless steel. Thus it was not possible to find a plastic-instability-failure equation based on specimen geometry.

REFERENCES

1. K. E. Horton, "Thermal-Stress-Fatigue Behavior of Stainless Steels," Quarterly Progress Report No. 6, ATL-A-137, EURAEC-756, April-June 1963.
2. J. G. Sessler and V. Weiss, "Low-Cycle Fatigue in Pressure-Vessel Materials," ASME Preprint 62, WA233, 1962.
3. L. F. Coffin, Jr., "A Study of the Effects of Cyclic-Thermal Stresses on a Ductile Metal," Trans. ASME, Series D, August 1954.

LIST OF SYMBOLS

A_f = final cross-sectional area (inches)

A_o = initial cross-sectional area (inches)

c, k = constants, material-dependent

N = cycles to failure

R = specimen mean radius (inches)

t = specimen wall thickness (inches)

T_m = mean temperature of testing ($^{\circ}\text{C}$)

$\Delta \epsilon_p$ = plastic strain range (in./in.-cycle)

$\Delta \epsilon_t$ = total strain range (in./in.-cycle)

REPORTS ISSUED SINCE INCEPTION OF PROGRAM

Quarterly Technical Progress Report, January-March 1962, ATL-A-130, EURAEC 336.
Quarterly Technical Progress Report, April-June 1962, ATL-A-131, EURAEC 335
Quarterly Technical Progress Report, July-September 1962, ATL-A-133, EURAEC 481
Quarterly Technical Progress Report, October-December 1962, ATL-A-134, EURAEC 555
Quarterly Technical Progress Report, January-March 1963, ATL-A-135, EURAEC 632
Topical Report, 10 May 1962, ATL-A-136, EURAEC 725
Quarterly Technical Progress Report, April-June 1963, ATL-A-137, EURAEC 756

* Report should have been dated 1963.

TABLE I
SUMMARY OF TESTS PERFORMED DURING THE QUARTER

(See Table II for Description of Test Conditions)

<u>Specimen No.</u>	<u>Test</u>
207	I. A. 1. a. <u>2</u> . c)-1d
216	I. A. 3. a. <u>3</u> . c)-*
217	I. A. 3. a. <u>3</u> . a)-**
250	IV. A. 1. a. <u>2</u> . c)-2b
251	IV. A. 1. a. <u>2</u> . c)-2c
252	IV. A. 1. c. <u>2</u> . a)-2b
253	IV. A. 1. a. <u>2</u> . a)-2b
254	IV. A. 1. a. <u>3</u> . c)-1c
255	IV. A. 1. a. <u>3</u> . c)-2b
256	IV. A. 1. a. <u>3</u> . a)-2b
257	IV. A. 1. a. <u>3</u> . c)-2c
258	IV. A. 1. a. <u>3</u> . a)-1c
259	IV. A. 1. c. <u>2</u> . a)-2c
260	IV. A. 1. a. <u>2</u> . c)-1c
261	IV. A. 1. a. <u>2</u> . a)-2c
262	IV. A. 1. a. <u>2</u> . a)-1c
263	IV. A. 1. c. <u>2</u> . a)-2c
264	IV. A. 1. a. <u>3</u> . a)-2c
273	IV. A. 1. a. <u>2</u> . c)-1b
274	IV. A. 1. a. <u>2</u> . e)-1b
275	IV. A. 1. a. <u>2</u> . c)-4c
276	IV. A. 1. a. <u>2</u> . c)-4b

* 650 to 150°C, 60-sec hold time.

** 675 to 125°C, zero hold time.

PROPOSED TESTING PROGRAM

PLASTIC STRAIN RANGE VERSUS CYCLES TO FAILURE FOR TSF-TESTED
AS-RECEIVED 304-L STAINLESS STEEL
(Short Measurement Gage Length. 60-Second Hold Time at Temperature Extremes.)

FIGURE 1

Neg. #2366

a. After 20 cycles

Neg. #2364

b. After 40 cycles

Neg. #2357

c. After 63 cycles

Neg. #2361

d. After 100 cycles

Neg. #2362

e. After 125 cycles

Neg. #2356

f. After 202 cycles

TYPE 304-L STAINLESS STEEL TSF-CYCLED BETWEEN 850 AND 350°C
 1-Min Hold at Temperature Extremes
 Taken at 850°C. Actual Specimen Diameter $\cong \frac{1}{2}$ "

FIGURE 2

PLASTIC STRAIN RANGE VERSUS CYCLES TO FAILURE FOR
304-L STAINLESS STEEL (COLD-WORKED) AND FOR AN INCOLOY ALLOY
(Short Measurement Gage Length. Tensile Test Ductility Obtained from $\ln(A_o/A_f)$)

FIGURE 3

Neg. #2387

PHOTOGRAPH SHOWING MACHINING DONE PRIOR TO WELDING ON
CROLOY $2\frac{1}{4}$ SEAMLESS PIPE AND 1018 CARBON STEEL END BOSSES

FIGURE 4

500×

Oblique Lighting

Neg. #4994

- a. As-received; Hardness, R_B 96-98; Electrolytic etch, 10% oxalic acid.

100×

Oblique Lighting

Neg. #4960

- b. Held 2 hours at 1000°C ($\sim 1832^\circ\text{F}$); Quenched in helium gas; Hardness, R_C 42-44; Etched in 10-HCl, 3-HNO₃, 100-CH₃OH.

TYPE 403 MARTENSITIC STAINLESS STEEL: a) ANNEALED; b) HARDENED

FIGURE 5

100×

Oblique Lighting

Neg. #4961

- a. Held 2 hours at 1000°C (1830°F); Quenched in helium;
Tempered 2 hours in vacuum at 649°C (1200°F);
Hardness, R_C 20-23; Etched in 10-HCl, 3-HNO₃,
100-CH₃OH.

100×

Oblique Lighting

Neg. #4993

- b. Same heat treatment as above except for 24-hour temper
at 649°C; Hardness, R_B 87-92; Same etch.

TYPE 403 MARTENSITIC STAINLESS STEEL: a) HARDENED AND TEMPERED 1 HOUR;
b) HARDENED AND TEMPERED 24 HOURS.

FIGURE 6

100×

Neg. #4954

500×

Neg. #4952

MICROSTRUCTURE OF A302B FERRITIC PRESSURE-VESSEL STEEL
(Etched in 4% Picral)

FIGURE 7

50×

Neg. #4953

UNETCHED SURFACE OF A302B FERRITIC PRESSURE-VESSEL STEEL
SHOWING SLAG INCLUSIONS
(Longitudinal Section. Region Taken from
 $1\frac{1}{4}$ " Diameter Bar at $\frac{1}{2}$ -Diameter Position.)

FIGURE 8

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

FIGURE 9

TOTAL STRAIN RANGE VERSUS CYCLES TO FAILURE FOR
A302B FERRITIC PRESSURE-VESSEL STEEL,

FIGURE 10

100×

← Stress →

Neg. #4978

- a. Specimen 253. Pure fatigue failure.
 $T_{\max} = 700^{\circ}\text{C}$; 0.030-inch wall; Unetched.

250×

← Stress →

Neg. #4988

- b. Specimen 256. Pure fatigue failure.
 $T_{\max} = 700^{\circ}\text{C}$; 0.040-inch wall; Hardness, R_B 86.

TSF-TESTED A302B FERRITIC PRESSURE-VESSEL STEEL
 SHOWING OXIDE GROWTH IN FATIGUE CRACKS

FIGURE 11

250×

← Stress →

Neg. #4982

- a. Specimen 262.
 $T_{\text{max}} = 600^{\circ}\text{C}$; Etched 1 min in 1% Nital.

1000×

← Stress →

Neg. #4984

- b. Same as above. "Black" areas pearlite;
 "White" areas ferrite.

TSF-TESTED A302B FERRITIC PRESSURE-VESSEL STEEL
 SHOWING OXIDE GROWTH

FIGURE 12

250× ← Stress → Neg. #5020

a. Showing etch gradient. Etched in 1% Nital for 1 min.

1000× ← Stress → Neg. #5021

b. Showing grain-size gradient. Same etch as above.

TSF-TESTED A302B FERRITIC PRESSURE-VESSEL STEEL
 RUN IN ARGON ATMOSPHERE WITH OXYGEN GETTERER OPERATIVE
 Specimen 263. Hardness, R_C 30 (center, not surface). $T_{max} = 800^\circ\text{C}$.

FIGURE 13

MICROHARDNESS INDENTATIONS ON SPECIMENS SHOWN
IN FIGURE 13

Note large indentation at surface (therefore, soft material)
and small indentations at center (therefore, hard material).

All indentations made with 50-gram load.

FIGURE 14

1000×

← Stress →

Neg. #4983

SPECIMEN 263 (SAME AS IN FIGURE 13) SHOWING
GRAIN ELONGATION

(Region near Center of Specimen Wall)

Etched in 1% Nital; Hardness, R_C 31.

FIGURE 15

250×

← Stress →

Neg. #4981

← Center of
Specimen
Wall

SPECIMEN 264, SHOWING GRAIN ELONGATION
(Specimen exhibited an instability failure.)

$T_{\max} = 800^{\circ}\text{C}$; Etched in 1% Nital; Hardness, R_C 32 (at center).

FIGURE 16

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