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American Radiator & Standard Sanitary Corporation
Advanced Technology Laboratories Division

INVESTIGATION OF
THERMAL-STRESS-FATIGUE BEHAVIOR
OF STAINLESS STEELS

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Mountain View, California

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INVESTIGATION OF
THERMAL-STRESS-FATIGUE BEHAVIOR
OF STAINLESS STEELS

Quarterly Progress Report No. 10

ATL Job 1230301

April-June 1964

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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

Testing of A302B ferritic pressure-vessel steel is complete, and testing of Types 403 martensitic stainless steel and A387D chromium-molybdenum (Croloy 2 $\frac{1}{4}$) steel is 50% completed.

Most of the tests conducted during this quarter were on A387D steel. Emphasis was placed, however, on vacuum tests of A302B steel in an effort to eliminate oxidation effects. Reverse thermal-stress-fatigue testing on 403 stainless steel was also emphasized in an effort to prevent the low-cycle buckling phenomenon.

A cursory electron-microscopic examination of tested specimens was made. The substructures of small- and large-grained materials were observed in order to ascertain why there was no grain-size effect on the thermal-stress fatigue behavior of 304-L steel.

The topical report on austenitic stainless steels (Types 304 and 304-L)¹ was submitted on April 13, 1964.

1. See list of references, page 11.

PRINCIPAL INVESTIGATORS

Investigators on the project include K. E. Horton, Project Leader, and J. M. Hallander, Assistant Metallurgist.

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 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 APRIL-JUNE 1964

A. Type A387D Steel (Croloy 2 $\frac{1}{4}$)

The thermal-stress-fatigue (TSF) and conventional, strain-cycling-fatigue (SCF) testing of Type A387D steel has progressed according to schedule. The results are plotted in Figure 1, from which it is seen that excellent data have been gathered to date. A noteworthy observation is the low fatigue resistance of this same steel found by Taira.² Although the slope of Taira's line is extremely low, it would be expected to be less than the slope of plastic strain range ($\Delta\epsilon_p$) because of the additive property of elastic and plastic strains. It is apparent that the temperature gradient along the measurement gage length of Taira's specimens resulted in an unrealistically low average value of strain measurement, illustrating the extreme importance of test technique. The method used herein to obtain $\Delta\epsilon_p$ and $\Delta\epsilon_t$ (total strain range) for the tests on A387D is the newer, graphical technique described in the previous quarterly progress report.³

The agreement between SCF (constant-temperature) and TSF tests in Figure 1 is indeed gratifying and was predicted prior to testing. This agreement is tentative, however, because the SCF test specimens have been failing at the ends of the uniform cross-sectional area, not in the instrumented maximum-temperature region. Since this undesirable failure has not occurred in any of the other alloys, specimens for which were machined from solid bars, it was concluded that the A387D specimens were weakened by the welding process used in fabricating them. Complicating the failure analysis is the fact that in no case has failure occurred in the weld metal or even in the heat-affected zone. This type of failure has been prevented in more recent SCF tests by a redesign of the test specimen which strengthened the specimen ends.

To date, no structural changes due to TSF or SCF testing have been found in this steel. The ferritic structure remained ferritic, and fatigue fractures were both trans-crystalline and intercrystalline. A typical transcrystalline fracture is shown in Figure 2, along with the untested structure for comparison.

B. Type 403 Martensitic Stainless Steel

TSF and SCF test results on Type 403 stainless steel (with a tempered-martensite structure) are plotted in Figure 3. Reverse thermal-stress-fatigue (RTSF) test* results

* Compression at low temperature, tension at high temperature.

are also shown on this graph. The tentative conclusions for this steel outlined in the previous quarterly progress report³ are still valid. Briefly, they are:

- 1) Increasing mean temperature of TSF testing decreases the resistance to alternating plastic strain.
- 2) Hold time at temperature extremes has no effect on fatigue resistance.
- 3) TSF and SCF test results are in agreement in that 400°C constant-temperature tests agree with 300, 400, 500, and 600°C mean-temperature TSF tests.

In addition to the above conclusions, there is a strong indication (Figure 3) of the existence of two failure lines, one for buckling and one for nonbuckling tests. The buckling line is, of course, the solid line on the left in Figure 3. The RTSF tests caused greater specimen instability than the TSF and SCF tests, as shown by the dashed line. This greater instability is a result of imposing far greater compressive loads on the specimen than were imposed during TSF tests. Even though these greater compressive loads are on the specimen when it is at the lower temperatures of cycling, rather than at the higher temperatures of cycling as on the TSF test specimens, the specimen still buckled in the very low cycles-to-failure range. In the higher cycles-to-failure range, the RTSF test results were identical to the TSF and SCF test results. These RTSF tests support the specimen behavior shown in Figure 4 and suggested in an earlier, special report.⁴

The tempering of the tempered martensitic structure during TSF testing caused the plastic strain range to increase during a test (ΔT being held constant). It is interesting to note that an annealed (as-received) structure also underwent an increase in plastic strain range during testing. This is not too surprising in view of the identical structures of as-received and TSF-tested tempered martensite, as shown in Figure 5.

The fractures of the martensitic steel have been transcrystalline, as shown in Figures 5b and 6.

C. Type A302B Ferritic Steel

The testing of this material was essentially completed with some vacuum tests directed toward eliminating oxidation effects. The results will be presented in the forthcoming final report.

D. Substructure Studies

The introduction of electron microscopy to metallurgical research in the last decade has enabled the visual observation of phenomena that previously had been only theoretical. Since it is possible to look through extremely small areas with the electron microscope, it is easily possible for microscopic heterogeneity to produce grossly different results in adjacent regions, thus making it difficult to draw conclusions.⁵ However, thorough examination by electron microscope of physically tested materials do yield unmistakable trends in substructure changes. For example, subgrains usually form in tensile- and fatigue-tested specimens, and it is generally found that subgrain size is independent of grain size.⁶ While subgrain size has been observed⁷ to vary inversely with total plastic tension strain, it has also been observed⁸ to vary directly with total plastic cyclic strain. Furthermore, at certain temperatures and after certain total plastic tension strain, subgrains have been observed to form but at lower temperatures and for less total plastic tension strain, subgrains do not form.⁷ Thus the effect of TSF testing on substructure was essentially unpredictable without experimentation. Accordingly, a brief transmission-electron-microscopic investigation was initiated on 304-L stainless steel, since this steel had been very thoroughly investigated.¹

The failed TSF test specimens were made into specimens for transmission electron microscopy by first using a hand grinder, cut-off wheel to cut a specimen coupon $3/8 \times 1$ " from the failure region. These coupons had a wall thickness of 20 mils and a radius of lateral curvature of $1/2$ inch. They were coated around the edges with an electrical insulating lacquer (to prevent preferential edge attack) and then made the anode in a solution of 60% sulfuric acid-40% phosphoric acid. A stainless-steel cathode in the form of two pointed wires, with $1/8$ inch between the points, was utilized to localize the electropolishing action to the center of the coupon. It was found that 1 to $1-1/2$ hours of electropolishing reduced the coupon thickness to 1 or 2 mils. Further polishing with a plate cathode brought regions of the coupon down to 1000 to 3000 angstroms thick, suitable for electron transmission.

In this initial work, it was decided to study the large- and small-grain-size specimens at both low and high cycles to failure. It will be recalled that a grain-size difference between annealed specimens did not produce an observable effect on TSF

resistance, based on plastic strain range or cycles to failure.¹ The relative TSF resistance of large- and small-grained 304-L stainless steel is shown in Figure 7, together with that of specimens examined with the electron microscope.

The substructure of a small-grained, low-cycles-to-failure specimen (No. 56) is shown in Figure 8. The subgrain boundaries are clearly delineated, along with dislocation tangles, dipoles, and extended loops. It is noticed that no stacking faults appear to be present, indicative of a rather high stacking fault energy. The lack of extended dislocation nodes also suggests a high stacking-fault energy. A stacking-fault energy range of 13 to 18 ergs/cm² has been quoted⁹ for this type of stainless steel.

The structure of the large-grained, low-cycles-to-failure specimen (No. 182) was rather difficult to assess in the time available for electron microscopy. There was a strong indication, however, of a more open substructure than in the small-grained material. That is, the subgrain size appeared larger in the large-grained material than in the small-grained material, as shown in Figure 9.

The substructure of a small-grained, high-cycles-to-failure specimen (No. 34) is shown in Figure 10. The subgrain size, which is approximately the same as in the low-cycles-to-failure specimen, is again clearly resolved, with a strong indication of greater misorientation between the subgrains than in the low-cycles-to-failure specimen. This increased misorientation is probably due to the increased total plastic strain relative to the low-cycles-to-failure test illustrated by Figure 8. Such an increase has also been suggested elsewhere.¹⁰

The substructure of the large-grained, high-cycles-to-failure specimen (No. 181) is shown in Figure 11. It is seen that this structure is also relatively open, as was the structure of the large-grained, low-cycles-to-failure specimen.

The foregoing substructure study was not thorough enough to permit tendencies to be firmly established. However, it may be tentatively concluded that subgrain size is not a function of total plastic strain, whereas it is a function of grain size. It is also probable that plastic strain greater than that necessary to form subgrains causes misorientation between subgrains to increase.

TSF AND SCF TEST RESULTS FOR TYPE A387D STEEL (CROLOY 2 $\frac{1}{4}$)

FIGURE 1

TSF, RTSF, AND SCF TEST RESULTS FOR
TYPE 403 MARTENSITIC STAINLESS STEEL

FIGURE 3

PROPOSED TSF (AND SCF) FAILURE LINES

FIGURE 4

Neg. #5183

a. As-received (annealed)

Neg. #5209

b. TSF-tested tempered martensitic specimen.
 $T_2 = 575^\circ\text{C}$, $T_1 = 225^\circ\text{C}$.

STRUCTURES OF 403 MARTENSITIC STAINLESS STEEL

Magnification: $250\times$

Etch: 3 Glycerol-2 HF-1 HNO_3

FIGURE 5

100×

← Stress →

Neg. #5556

TSF-TESTED 403 STAINLESS STEEL
 $T_2 = 500^{\circ}\text{C}$, $T_1 = 100^{\circ}\text{C}$.

FIGURE 6

PLASTIC STRAIN RANGE VERSUS CYCLES TO FAILURE
 FOR LARGE-GRAIN-SIZE (ASTM 1) AND SMALL-GRAIN-SIZE (ASTM 6-7)
 TSF-TESTED 304-L STAINLESS STEEL

FIGURE 7

35,500×

Neg. #1658b

15,700×

Neg. #1657b

SUBSTRUCTURE OF SMALL-GRAINED, LOW-CYCLES-TO-FAILURE
304-L STAINLESS STEEL (SPECIMEN 56)

FIGURE 8

26,100×

Neg. #1603b

**SUBSTRUCTURE OF LARGE-GRAINED, LOW-CYCLES-TO-FAILURE
304-L STAINLESS STEEL (SPECIMEN 182)**
Note that dislocations lie primarily in two slip planes.

FIGURE 9

31,800×

Neg. #1751a

24,800×

Neg. #1751b

SUBSTRUCTURE OF SMALL-GRAINED, HIGH-CYCLES-TO-FAILURE
304-L STAINLESS STEEL (SPECIMEN 34)
Subgrain size is approximately the same as in
the low-cycles-to-failure specimen (Figure 8).

FIGURE 10

54,375×

Neg. #1784a

SUBSTRUCTURE OF LARGE-GRAINED, HIGH-CYCLES-TO-FAILURE
304-L STAINLESS STEEL (SPECIMEN 181)
Subgrains not delineated.

FIGURE 11

PLANS FOR FUTURE WORK

The testing of A387D (Croloy 2 $\frac{1}{4}$) will continue, with emphasis being placed on studying the effect of strain rate (rate of temperature change). Reverse-thermal-stress-fatigue tests will be continued on 403 martensitic stainless steel. A cursory TSF test program will be conducted on zirconium, with the aim of comparing long and short measurement gage length data. This metal (as well as Zircaloy II) was tested in a previous research program at ATL¹¹ but at a time when test techniques were inferior to those in use now. Much of the previous data on zirconium will be made accurate by applying the strain-range conversion factors to be found during the next quarter. It should be emphasized that the earlier data are correct when relative results are considered.

All testing will be completed by the end of the next quarter. Subsequently, a final report summarizing work done on all alloy systems will be written.

CONCLUSIONS

1) Results of conventional strain-cycling-fatigue and thermal-stress-fatigue tests agree when compared on the basis of plastic strain range versus cycles to failure.

However, the agreement is obtainable only under certain SCF test conditions.

2) Reverse-thermal-stress-fatigue test conditions cause greater specimen instability in the low-cycles-to-failure region. In the high-cycles-to-failure region, the normal and reverse TSF test results are in agreement.

3) Substructure studies of TSF-tested specimens of different grain size yielded tentative results indicating that subgrain size is proportional to grain size and that the angle of misorientation between subgrains is proportional to the total plastic strain experienced by the specimen.

LIST OF SYMBOLS

N = cycles to failure

T_1 = lowest temperature of TSF testing

T_2 = highest temperature of TSF testing

ΔT = temperature range during TSF testing ($T_2 - T_1$)

$\Delta \epsilon_p$ = plastic strain range

$\Delta \epsilon_t$ = total strain range

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